THE APPLICATION OF NEUTRON ACTIVATION ANALYSIS AND NUMERICAL TAXONOMIC METHODS TO THE STUDY OF ANCIENT FINE GREY POTTERY (*)

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ABSTRACT - The concentrations of three major elements and fourteen trace elements in selected sherds of Iron Age and first century A.D. fine grey ceramics, found at five different archaeological sites in Portugal, were determined by instrumental neutron activation analysis. These concentrations were used to calculate distance matrices which where then subjected to cluster analysis employing SAHN methods, k-means cluster analysis and principal components analysis, to group the sherds according to the overall similarity of their compositional features. The results showed that roughly equivalent groupings appeared regardless of the numerical method used. Three major clusters were recognized corresponding to three different sites, namely Conímbriga, Santa Olaia and Lisbon, suggesting that the ceramics from these sites were made locally. Some of the sherds studied showed patterns suggesting trade relationships between sites.

1-INTRODUCTION

At Conímbriga, as well as at other archaeological sites in continental Portugal, large quantities of sherds of dark-grey pottery have been recovered, which possess the common feature of being very carefully made. Although mineralogical analysis has been unsuccessful to discriminate between those sherds, archaeologists were able to define two classes based on traditional archaeological attributes (shape, finishing and presence in a particular stratigraphic level), namely: (i) Iron Age fine grey pottery and (ii) Roman fine grey pottery dating from the first century A. D. [1].

^(*) Results presented at the Conference of the Portuguese Physics Society (Lisbon, February 1978).

Important archaeological questions concerned with the study of this pottery are, on the one hand, whether it is possible to define local centers of manufacture and, on the other, whether one is able to establish for each burial deposit any relationships between the Iron Age grey ware and the Roman grey ceramics.

To answer this type of question, activation analysis has been frequently used and the analytical data processed by methods of numerical taxonomy [2].

In the present paper we report the results of the application of instrumental neutron activation analysis and of some numerical taxonomic methods to a group of sherds of this fine grey pottery, which were made available by Drs. A. Alarcão and J. Alarcão of the *Museu Monográfico de Conímbriga*. This contribution is the first part of a study aimed at answering the questions referred to above.

2 — EXPERIMENTAL

Analysed sherds. The provenances and identifications of the sherds studied are presented in table 1.

Sherd	Provenance	Class according to archaeological evidence
C 16 $-$ C 24 C 26 C 28 $-$ C 32	Conímbriga	Iron Age
C 25, C 27 C 33 – C 36	Conímbriga	Roman (first century A. D.)
F 37	Fiães (Feira)	3)
SO 1 – SO 9	Santa Olaia (Montemor·o-Velho)	Iron Age
TA 10	Tavarede (Figueira da Foz)	3
L 11 – L 15	Lisbon	>

TABLE 1 - Provenances and identifications of sherds

Sample preparation. The sherds were sampled by using the procedure employed by Abascal, Harbottle and Sayre [3]. A fraction of about 1 g was usually removed from each sherd. Each of those fractions was then carefully homogenized by mixing, and oven-dried at 110°C before use. Samples of about 100 mg of each homogenized powder were finally weighed out and sealed into ultrahigh purity containers. The samples for short irradiations were introduced into small polyethylene cans. Those for long irradiations into fused quartz ampoules.

Standards. Six U.S. Geological Survey standard rocks, namely G-2, BCR-1, GSP-1, AGV-1, PCC-1 and DTS-1, were used as standards. Weighed amounts of the first four, dried and packaged in the same fashion as the ceramic samples, were included with each set of 16-20 cans in the short irradiations. Weighed amounts of all six, prepared in the same manner, were included with each set of 30-35 ampoules in the long irradiations.

Irradiations. All irradiations were done in the core grid of the RPI reactor (Sacavém) at a neutron flux of $\sim 3.5 \times 10^{12}$ n.cm⁻².s⁻¹. For each sample two irradiations were made: a 10 minutes one and a 70 hours one (14 hours periods on 5 successive days). Flux monitors were irradiated together in order to measure differences in neutron fluxes at the positions where samples and standards stood.

Counting. The equipment used for measurements was a gammaray spectrometer consisting of a 54 cm³ Ge(Li) coaxial detector (Ortec) connected through an Ortec model 120-4 F preamplifier and an Ortec 452 amplifier to an Intertechnique Didac 4000 multichannel analyser. This system had a FWHM resolution of 2.5 keV at 1.33 MeV energy. The spectrometric data were collected on punched paper tape for further processing. Since the polyethylene cans and silica tubes contribute no significant blank, samples and standards were left in their irradiation containers for radioassay. Measurements of the gamma spectra were made at four decay times: 2 hours after the short irradiation, for counting ⁵⁶Mn; 20 hours after the same irradiation, for counting ¹⁴⁰La and ¹⁷⁷Lu; and 28-35 days after this

Portgal Phys. - Vol. 10, fasc. 1-2, pp. 101-116, 1979

103

irradiation, for counting ¹⁴¹Ce, ²³³Pa, ⁵¹Cr, ¹⁸¹Hf, ¹³¹Ba, ¹³⁴Cs, ⁴⁶Sc, ⁸⁶Rb, ¹⁸²Ta, ⁵⁹Fe, ⁶⁰Co and ¹⁵²Eu. The gamma-ray energies used in the determination of each element are given in table 2.

Element	Radionuclide	Energy of gamma-rays used (keV)
Sodium	24 Na	1368
Potassium	42 K	1525
Rubidium	⁸⁶ Rb	1077
Cesium	134 Cs	796
Baríum	131 Ba	496
Scandium	46 Sc	889
Lanthanum	140 La	1596
Cerium	¹⁴¹ Ce	145
Europium	¹⁵² Eu	1408
Lutetium	177 Lu	208
Hafnium	181 Hf	482
Thorium	233 Pa	312
Tantalum	182 Ta	1221
Chromium	51 Cr	320
Manganese	⁵⁶ Mn	847
Iron	⁵⁹ Fe	1099
Cobalt	60 Co	1332

TABLE 2 — Radionuclides and energies of gamma-rays used in the determination of element concentrations

Spectrum analysis. All spectra were processed in a PDP 15 computer using computer programs which are adaptations of the GELIAN and OLIVE programs [4], [5]. The values of each element in each standard rock used for calculating element concentrations in samples were taken from data tabulated by Abascal, Harbottle and Sayre [3].

3-METHODS OF DATA ANALYSIS

Data matrix. The data used in this study are the results obtained by applying the instrumental neutron activation analysis method described above, which are shown in table 3. Thus our original data matrix has 37 columns representing the 37 sherds (OTU's) to be grouped, and 17 rows representing the concentrations of the 17 elements (attributes) which have been analysed in those sherds.

Scaling. Standardization of attributes was applied to the raw data in order to equalize the size of each attribute. In this method one computes the mean and standard deviation of the states of each attribute and expresses each state as a deviation from the mean in standard deviation units. Logarithmic transformation was also applied for the purpose of seeing how the final classification would be affected by the scaling method.

Measures of similarity. Two dissimilarity coefficients were calculated, namely the mean Euclidean distance, d_{jk} , and the mean character difference, $(MCD)_{jk}$, defined as follows:

$$d_{jk} = \left[\frac{1}{n} \sum_{i=1}^{n} (\mathbf{X}_{ij} - \mathbf{X}_{ik})^2\right]^{1/2}$$
$$(\text{MCD})_{jk} = \frac{1}{n} \sum_{i=1}^{n} |\mathbf{X}_{ij} - \mathbf{X}_{ik}|$$

where *n* is the number of attributes, X_{ij} is the transformed value of OTU *j* for attibute *i* and X_{ik} is the transformed value of OTU *k* for the same attribute.

Cluster analysis. This analysis was carried out by various methods: some SAHN methods (complete linkage, UPGMA and size-of-cluster methods) and a nonhierarchic method (k-means). The results of SAHN methods were represented by phenograms. Cophenetic correlation coefficients between the dissimilarity values implied by the phenograms and those of the original dissimilarity

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106

	C16	C17	C18	C19	C20	C21	C22	C23	C24	C25	C26	C27
												-
Ja20 %	0,240	0,164	0,198	0,324	0,712	0,259	0,303	0,204	0,192	0,205	0,195	0,195
20 %	3,95	4,04	3,73	4,32	3,73	4,28	4,64	3,99	4,11	3,70	4,48	4,34
(b2O	295	356	331	324	248	335	320	330	349	328	354	388
520	24	33	26	25	22	26	24	29	31	24	31	30
B aO	456	512	564	490	266	417	582	683	602	475	568	518
c.203	20	18	18	25	30	14	14	17	18	16	16	17
.a2O3	87	70	68	84	65	18	65	72	76	67	73	57
ceO2	156	114	130	145	128	194	135	125	145	110	130	224
cu2O3	1,7	1,5	1,6	2,3	2,3	1,4	1,4	1,6	1,8	1,1	1,6	2,5
.u2O3	0,99	0,84	16'0	66'0	1,27	66'0	0,86	0,86	66'0	0,81	0,88	1,02
HO2	10,1	9,2	9,3	10,0	7,0	14,5	9,8	9,4	9,6	9,5	10,5	12,4
ChO ₂	33,7	34,8	34,8	33,4	20,4	47,7	34,2	35,0	36,3	39,6	38,8	52,5
.a205	3,9	5,5	6,0	4,3	2,9	6,9	1,9	5,7	6,3	5,7	5,7	5,8
Cr2O3	84,0	66,5	68,4	110	132	45,6	48,8	61,7	59,5	49,3	47,1	51,5
Ouh	169	217	274	358	261	403	184	240	279	156	202	371
:e2O3%	4,42	4,53	5,00	6,22	3,81	5,50	3,92	4,71	4,60	3,87	4,83	69,69
000	6	7	2	17	20	17	11	17	24	14	9	0

Portgal Phys. - Vol. 10, fasc. 1-2, pp. 101-116, 1979

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J. M. P. CABRAL et al. - Neutron activation analysis of ancient pottery

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	C28	C29	C30	C31	C32	C33	C34	C35	C36	F37	S01	S02
Na20%	0,411	0,219	0,208	0,231	0,210	0,212	0,260	0,190	0,178	0,150	0,349	0,603
K2O %	4,05	3,78	3,71	4,39	4,29	4,67	3,89	4,07	4,18	3,58	3,44	3,90
Rb2O	286	325	312	318	351	379	783	352	355	297	267	230
Cs2O	23	30	25	26	32	30	54	30	30	25	24	21
BaO	670	502	588	478	497	552	1520	396	521	406	496	690
Sc2O3	16	16	16	16	18	17	37	19	18	18	27	25
La2O3	19	77	65	19	65	92	63	16	76	17	60	64
CeO ₂	130	152	131	135	130	164	282	214	149	110	129	109
Eu2O3	1,4	1,7	1,5	1,4	1,6	2,2	2,9	2,1	1,5	1,5	2,2	2,0
Lu2O3	0,77	0,95	0,79	0,76	0,87	1,04	0,73	1,05	0,79	0,77	0,81	1,03
HfO ₂	7,5	12,1	9,1	9,8	9,6	11,5	21,5	11,7	9,7	10,4	7,3	8,0
ThO ₂	30,9	42,8	32,7	36,5	36,7	44,3	85,5	46,4	43,8	47,0	20,6	21,3
Ta2O5	4,8	5,9	6,1	5,1	6,0	5,8	11,4	6'9	6,1	6,2	2,9	2,9
Cr2O3	59,1	55,4	70,7	61,5	70,5	57,2	139	75,3	54,7	60,6	116	105
MnO	252	195	231	297	214	251	245	359	273	148	199	201
Fe2O3%	4,19	6,07	4,55	4,59	5,21	5,12	11,7	5,56	6,21	6,23	4,58	3,73
CoO	13	14	30	6	9	10	15	п	10	7	13	13

Portgal Phys. - Vol. 10, fasc. 1-2, pp. 101-116, 1979

107

J. M. P. CABRAL et al. - Neutron activation analysis of ancient pottery

TABLE 3-Oxide concentrations for specimens of Iron Age and early-Roman sherds from Conímbriga, Santa Olaia, Fiães (Feira), Tavarede and Lisbon

	S03	S04	S05	S06	S07	S08	S09	TA10	TII	L12	L13	L14	L15
%0%	0,623	0,728	0,435	0,427	0,335	0,595	0,591	0,158	0,259	0,851	1,10	0,819	1,06
%C	3,54	4,15	2,98	3,36	4,85	3,81	4,42	3,25	4,02	4,04	3,43	4,02	4,81
0	242	241	215	249	377	255	285	205	301	243	198	242	248
0	20	22	14	22	25	22	32	25	39	14	11	13	15
0	746	888	788	563	669	872	554	566	577	740	467	736	617
03	22	27	18	24	15	28	23	26	26	31	24	31	31
03	29	70	50	64	67	19	51	47	56	40	38	40	49
02	139	118	96,4	104	160	121	112	82,9	116	83,5	86,2	0,67	117
03	2,0	2,3	1,7	2,1	1,6	2,2	1,6	1,8	2,3	1.7	1,6	1,6	2,3
03	0,96	1,06	0,77	0,95	0,81	0,99	0,62	0,74	06'0	0,67	0,64	0,68	0,76
20	1'6	7,6	8,2	8,9	11,4	8,2	6,9	6,3	6,4	5,6	5,7	5,7	6,0
02	23,3	22,9	16,6	21,8	37,9	22,0	23,1	20,8	23,1	15,1	13,8	15,5	16,6
05	3,1	2,9	2,4	2,9	5,3	3,1	3,2	3,6	3,5	2,0	2,4	2,0	2,4
03	90'0	116	73,9	101	45,3	124	88,0	106	104	148	123	146	151
0	209	283	209	191	202	166	332	162	194	192	328	169	241
03%	3,68	3,55	3,52	4,21	4,32	4,07	3,50	5,67	4,92	5,33	5,43	5,31	5,85
0	15	14	12	14	80	12	11	35	29	16	21	14	26

J. M. P. CABRAL et al. - Neutron activation analysis of ancient pottery

108

matrices were calculated. The k-means method was applied repeated times for partitions into k clusters from k = 2 to k = 7.

Ordination methods. Ordination of samples in A-space was carried out by employing principal components analysis. The variation accounted for by a given number of principal components as well as the cophenetic correlation between distances in the three tirst principal components space and the fully dimensional one were calculated.

Computations. They were carried out by employing the MINT computer programs system [6] as well as the following programs: NADIST [7], AGCLUS [8] and BUILD [9]. These programs were run either at the computer center of the *Instituto Gulbenkian de Ciências*, *Oeiras*, or at the computer center of the Universities of Lisbon.

4-RESULTS AND DISCUSSION

Fig. 1 shows the results of the UPGMA clustering obtained with the standardized data and using the mean Euclidean distance as a measure of dissimilarity. The cophenetic correlation coefficient was 0.91 indicating a good agreement of the phenogram with the original dissimilarity matrix. A very good agreement was also found between the phenogram illustrated in fig. 1 and the result of the UPGMA clustering based on log transformed data and the same distance measure. This observation confirms an earlier conclusion [10] that change of scaling method does not seem to affect significantly the clustering of archaeological ceramics.

The results of the other mentioned hierarchic methods obtained with the identically transformed data and the same dissimilarity measure did not reveal any important disagreement with the result presented in fig. 1. Moreover, it was found that change of the distance coefficient did not produce any significant effect on the classification. It appears therefore that the phenogram of fig. 1 can be considered as a good representation of the taxonomic structure no matter which scaling method, distance measure and SAHN method of clustering has been employed.

This phenogram suggests that the Conímbriga samples, Santa Olaia samples and Lisbon samples may be compositionally distingui-

shable. In fact four clusters are discerned, two composed of practically all Conímbriga sherds, another composed primarily of Santa Olaia sherds and the fourth composed of nearly all Lisbon sherds. Most of Iron Age Conímbriga sherds (twelve out of fourteen) are in one cluster.



DISTANCE

Fig. 1 — Phenogram of 37 sherds of fine grey ceramics based ou UPGMA cluster analysis of mean Euclidean distances. The cophenetic correlation coefficient was 0.91

Three of the six first century A.D. Conímbriga sherds are in the other. It is also shown that one of these sherds (C 34) is an outlier, another (C 20) belongs to the Santa Olaia cluster and that, inversely, a Santa Olaia sherd (SO 7) pertains to one of the Conímbriga clusters.

In addition, the phenogram indicates that the only sherd from Fiães is included in this Conímbriga cluster and that the only sherd from Tavarede, as well as one of the Lisbon samples (L 11), may be members of the Santa Olaia cluster.

The results of the k-means analysis are presented in table 4. Since C 34 sample was previously identified as an outlier, it was not considered in this analysis. These results confirm most of the conclusions from SAHN methods of cluster analysis. As a matter of fact the set of sherds was first split into two basic groups with all Conímbriga sherds (except C 20), the SO 7 and F 37 samples in one, and the residue in the other. It should be pointed out that the group of Conímbriga sherds always remained separated in all further subdivisions. This result suggests that probably the two clusters of Conímbriga sherds found by using SAHN methods of clustering are not significantly different. At the three-cluster partition the residue was split into two groups, one composed mainly of Santa Olaia samples (except SO 5, SO 7 and SO 9), the C 20 and L11 samples, and the other of the residual ones. The four-cluster partition divided the second residue into two groups, one consisting of three Lisbon samples and the other of the SO 5, TA 10 and L 13 samples. At the five-cluster partition there was no split and only a very slight rearrangement of samples was observed, which consisted in the attachment of L 13 sample to the group of Lisbon sherds and of the TA 10 sample to the group of Santa Olaia sherds, so that the SO 5 sample remained alone in one group. The six-cluster and seven cluster partitions divided the group of Santa Olaia sherds into subgroups, leaving the Conímbriga cluster, the Lisbon cluster and the SO 5 sample as they were at the five-cluster level.

In order to know how many clusters should be considered the values of the reduction of within-cluster variance for each attribute between the k-cluster and (k+1)-cluster partitions, measured by means of the appropriate «F-ratio», as well as the values of the overall mean square ratio, were compared [9]. Those values indicate that partitions with k > 5 are not justifiable.

The results of ordination by principal components analysis are presented in fig. 2. The relative contribution of the original attributes to the first three principal components, as well as the percentages of the total variance accounted for by them, are shown in table 5. It should be pointed out that, as in the case of the application of k-means algorithm, the C 34 sample was not considered in this ana-

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TABLE

Number of clusters	Error of partition		Denotation and composit	tion of clusters	
k=5	271.2	CC-Conimbriga cluster All Conimbriga sherds (except C20) + S07 + + F 37	SOC-Santa Olaia cluster All Santa Olaia sherds (except SO5 and SO 7)+ + C 20 + TA 10 + L 11	SO 5	LC-Lisbou cluster All Lisbon sherds (except L 11)
k=4	281.1	CC	SOC-TA10	SO 5 + TA 10 + L 13	LC-L13
k = 3	311.7	CC	SOC-(TA10+SO9)	SO 5 + SO 9 +	TA 10+LC
k=2	361.6	cc		soc+sos+Lc	
k = 1	612.0		cc + soc + so	05+LC	

Portgal Phys. --- Vol. 10, fasc. 1-2, pp. 101-116, 1979

112

J M. P. CABRAL et al. - Neutron activation analysis of ancient pottery



J. M. P. CABRAL et al. - Neutron activation analysis of ancient pottery

Fig. 2 — Ordination plots of 36 sherds of fine grey ceramics from principal components analysis. Signs and letters denote findspots: ● C (Conímbriga); ■ SO (Santa Olaia);
TA (Tavarede) and F (Fiães); ▲ L (Lisbon). Closed signs denote Iron Age ceramics, open ones represent first century A. D. ceramics.

Portgal Phys. - Vol. 10, fasc. 1-2, pp. 101-116, 1979

113

10-2-4-2		Principal component	8
Attributes	1	2	3
1 No O %	0.997	0.168	0.000
2 K 0 %	-0.301	-0.100	0.028
3 RbsO nnm	-0.913	-0.210	-0.223
4 CsoO	-0.719	-0.094	-0.124
5 BaO	0.623	-0.377	0.374
6 Sc.O. »	0.839	- 0 387	-0.294
7 La ₂ O ₃ »	-0.840	-0.451	0.054
8 CeO ₂ »	-0.777	-0.476	0.031
9 Eu ₂ O ₃ *	0.249	-0.890	- 0,128
10 Lu ₂ O _{3 *}	-0.298	-0.807	0.254
11 HfO ₂ ,	- 0.908	-0.139	0.151
12 ThO ₂ »	-0.972	-0.019	-0.082
13 TagO ₅ »	-0.934	0.152	-0.072
.14 Cr ₂ O ₃ *	0.884	-0.316	-0.254
15 MnO »	-0.057	-0.086	0.510
16 Fe ₂ O ₃ %	- 0.313	-0.083	-0.739
17 CoO ppm	0.427	-0 .055	-0.386
elative importance of components adicated by			
igenvalue	8.446	2.410	1.471
6 variance	49.7	14.2	8.65
umulative % variance	49.7	63.9	72,5

TABLE 5 — Relative contribution of attributes to first principal components and relative importance of these components

lysis. These results are a most useful complement to the former's. On the one hand, they provide a very satisfactory overall view of relationships between samples in three dimensions (the cophenetic correlation coefficient between the distances implied in the three-dimensional ordination and the original distance matrix is equal to 0.966 and the proportion of the variation accounted for by the three dimensions is equal to 72.5%). On the other, they provide very

useful information by themselves, particularly as far as the attribute weights on principal components are concerned. In this study the first component, accounting for 49.7% of the variance, may be seen from table 5 to be appreciably related to several attributes, but most markedly to sodium, rubidium, scandium, lanthanum, hafnium, thorium, tantalum and chromium concentrations. The second component, accounting for 14.2% of the variance, is heavily influenced by two attributes: europium and lutecium concentrations. The third component, accounting for 8.7% of the variance, is mainly influenced by the iron concentration.

As seen in fig. 2 the results of ordination by principal components analysis also confirm most of the conclusions from SAHN methods of cluster analysis.

5 - CONCLUSIONS

Activation analysis and numerical taxonomy were applied to study 37 sherds of dark-grey pottery, 30 of the Iron Age, found at Conímbriga, Santa Olaia, Tavarede and Lisbon, and seven of the first century A.D., found at Conímbriga and Fiães. The study of this pottery has posed some questions to archaeologists, in particular whether one centre of manufacture supplied those and other sites or they were made at the sites, and whether it is possible to establish any relationships between ceramics of both periods.

The results obtained show that roughly equivalent groups are produced regardless of the numerical taxonomic method used. Three major clusters are recognized corresponding to three different sites, namely Conímbriga, Santa Olaia and Lisbon. The answer to the first question is then clear, that the sherds from these three sites were very likely made at the sites. However, doubts still subsist as far as Tavarede and Fiães sherds are concerned, since only one specimen from each site was analysed. Moreover, the results indicate that Conímbriga and Santa Olaia clusters include at least one sherd found at Santa Olaia and Conímbriga, respectively, suggesting trade relationships between sites. The results show in addition that, with the exception of one sherd (C 34), there are probably no significant differences in compositional patterns between Iron Age and first century A. D. Conímbriga sherds.

Portgal Phys. - Vol. 10, fasc. 1-2, pp. 101-116, 1979

115

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