

INSTRUMENTAL NEUTRON ACTIVATION ANALYSIS OF POTTERY AND CLAY FROM THE ZURRABAH KILN COMPLEX*

by
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Twenty two sherds from the kiln complex at Zurrabah (excavated in 1980-81 under the direction of Dr. Fawzi Zayadine; Zayadine, 1981; 1982; 1986), and seven clay samples from two back rooms of the complex were analysed by neutron activation analysis. The sherds are described in Table 1. Most of the sherds are kiln wasters, ZP2 is of particular interest: its 'slip' is most probably due to a material in the body of the vessel which migrated to the surface when the vessel was drying. The extension of the 'slip' onto the breakage section indicates it broke either before, or at the start of, the firing, when the breakage section would have acted like any other surface to the migrating material.

The sherds were prepared for analysis by scraping clean a small area of a surface using a rough piece of synthetic sapphire. The rest of the surfaces were then covered with masking tape, leaving only the cleaned area clear. A further layer of that area was then removed using a synthetic sapphire drill head mounted on a small hand drill, then approx. 150mg of powder were drilled out from the centre of the sherd. The clay samples contained a few fragments of marine bi-valve fossils, indicating that they had not been levigated in antiquity. Other than that, the clay samples seemed of quite high purity, and, due to the fact that it is not known as to what extent the ancient Nabataean potters levigated their clays, it was decided not to subject the clay samples to any treatment except for drying prior to the analysis. The dried clay was then crushed to powder between polyethylene sheets.

Around 100mg of the powder from each sample were weighed into polyethylene irradiation capsules. The samples

were irradiated in several batches. Each batch had ten samples and two irradiation capsules containing the 'internal' standard, placed in outer 'core' tubes. The 'internal' standard was prepared from pottery sherds at the London Institute of Archaeology and calibrated against the IAEA's soil-5 standard.

The irradiation and counting were carried out at the University of London Reactor Centre in Ascot. The batches were irradiated at the core of the reactor, where the thermal flux is about $1.33 \times 10^{12} \text{ ncm}^{-2} \text{ s}^{-1}$, for four "working" days. At the reactor centre this is 7-8 hours a day with 16-17 hours during which the reactor is turned off.

The counting was carried out six days after the end of each irradiation, using a fully automated system incorporating a germanium-drifted lithium - Ge(Li) - detector with a sample changer, coupled to an ND6620 multi-channel analyser. Each sample was counted for one hour and thirty minutes (5400 seconds).

Fourteen elements were determined. Their concentrations in the samples are given in Tables 2 and 3. Of these fourteen elements, Na, Rb, and Eu proved unreliable, and are therefore not included in the statistical analyses of the results ('Amr, 1986). For the data analysis, the statistical package GENSTAT was used (Alvey et al., 1983). It was submitted from remote-entry terminals to the Cambridge University IBM 370/165 machine.

To separate the samples into clusters, the k-means method, using the sum of squares criterion, was employed (Doran and Hodson, 1975). What the method basically does is that it splits the units into a given number of clusters, calculates the

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centroids of all the clusters, then reshuffles the units until an optimum criterion value is reached. In this case when the total sum of squared Euclidean distances between each unit and the centroid of the cluster to which it is allocated is minimised. This is equivalent to maximising the sum of the squared distances between the cluster centroids.

The Zurrahah samples were subjected to the k-means method, starting at $k=7$ and going down to $k=2$. The log-transformed values were used as most of the elements investigated proved to have (roughly) log-normal distributions ('Amr, 1986). The elemental concentrations were also divided by their standard deviations in the samples in order to give the elements equal weights. The plot of the criterion value vs. the number of clusters (Fig. 1) shows that most of the decrease in the criterion value is accounted for at $k=5$. This is an indication that there are five clusters present. The classification at $k=5$ is given in Table 4.

To test the separation of the clusters, canonical variate analysis was used. In canonical variate analysis, the units are projected into a new space having fewer dimensions than the original space (the original space being defined by a number of dimensions equal to the number of given attributes). The new dimensions (called canonical variates) are correlated with the original dimensions in accordance to their effect in discriminating between the given clusters. In the majority of cases the first two or three canonical variates account for most of the variance. The elemental concentrations were log-transformed and then subjected to the canonical variate analysis. The plot of the first two canonical variates are in Fig. 2. The first canonical variate accounts for 91.6% of the variance, and the second 7.5%, totaling 99.1% of the variance. The '+'s in the plot denote the centres of the clusters, and the ':'s indicate where more than one value coincide. Clusters 1 and 2 are somewhat close in the plot. All the members of cluster

2 have concentration values for all the elements smaller than the values for cluster 1, and when the mean concentrations for cluster 2 are divided by the means for cluster 1, a factor of around 0.75 is obtained (Table 5). This indicates that both composition groups represent the same origin, with more 'inert' temper (e.g. silicates or calcites) being present in the fabric of the samples of cluster 2, all of which are coarse wares. Added to this is the fact that the number of samples studied is somewhat small, and both the k-means method and the canonical variate analysis assume all the clusters to have similar variances, which is not the case. It is felt that clusters 1 and 2 are 'artificially' separated, they seem to represent one composition group and should be amalgamated into one single cluster.

The final classification is given in Table 6, where all the pottery samples, except for ZP2, form one composition group. The clay samples are separated into two other composition groups. The composition of the final composition groups is given in Table 7. The plot of the first two canonical variates are in Fig. 3. The first canonical variate accounts for 85.0% of the variance, and the second 14.5%, totaling 99.5% of the variance.

ZP2 is definitely a waster, its differing composition is most probably due to the added material responsible for the 'slip' on its surface. It has La, Ce, Lu and Sm concentrations substantially higher than any of the other samples. None of the clay samples match the pottery. The clays were not levigated and therefore may have just been used as flooring material in the back rooms.

In conclusion, it seems that the Zurrahah potters used the same clays for making different types of pottery, although different manufacturing processes were involved. Most probably the clays found in the workshop were not used for making the pottery.

Khairieh 'Amr

Table 1 Description of the pottery samples

<i>Sample</i>	<i>Prov.</i>	<i>Vessel</i>	<i>Ware description</i>
ZP1	A. 1. 14	Cooking pot, ribbed.	Black (over-fired). Dark brown core. Many small white grits. Traces of green-creamy slip.
ZP2	A. 1. 14	Holemouth jar, collared rim.	Creamy pink. Some small-medium white and grey grits. Deep reddish-brown 'slip' on both surfaces, extending on part of one breakage section. Slightly encrusted.
ZP3	A. 1. 15	Fine painted bowl (late Nabataean).	Red-brown. Wide black core. Few small white grits. Very dark grey paint.
ZP4	A. 1. 15	Four body sherds of thin walled bowls melted and fused together in one mass.	Black - deep wine red - brown (over-fired), with a few 'bubbles'.
ZP5	A. 2. 6	Cooking pot.	Light red. Some small white and grey grits. Traces of brown slip. Heavily encrusted.
ZP6	A. 2. 8	Handle.	Yellow buff. Pinkish core. Some small white and grey grits. Brown slip.
ZP7	A. 2. 8	Jar.	Light pink. Light buff-grey core. Some large white and grey grits. Brown-grey slip. Encrusted.
ZP8	A. 2. 9	Fine Nabataean body sherd.	Red. Few small white and grey grits. Deeper red slip on the outer surface.
ZP9	A. 2. 10	Fine painted bowl (late Nabataean).	Red. Some small white and grey grits. Traces of dark brown paint.
ZP10	A. 2. 14	Fine painted bowl (late Nabataean).	Red. Thin buff-grey core. Few small white grits. Dark brown paint.
ZP11	A. 2. 14	Fine Nabataean closed bowl.	Red. Some small white and grey grits. Deeper red slip on the outer surface.
ZP12	A. 3. 15	Thin walled open bowl.	Grey-black (over-fired). Wide brown core. Some small white and grey grits. Turning yellowish in bands on the inner surface and part of the outer surface, with grey showing in the grooves of the wheel-marks. Yellow extending on some of the breakage sections.
ZP13	A. 3. 15	Fine painted bowl (late Nabataean).	Red. Wide black core. Some small white and grey grits. Dark brown paint.
ZP14	A. 3. 19	Vertical rimmed open fine Nabataean bowl.	Red. some small white and grey grits.
ZP15	A. 3. 19	Jar.	Buff-brown. Grey core not showing at the breakage sections. Many small-medium white and black grits, giving a mottled appearance. Traces of buff slip.
ZP16	A. 4. 2	Jar.	Light red. Grey core. Some small white and grey grits.
ZP17	A. 9. 8. 11 (436)	Four sherds from thin walled bowls, melted, fused together, and greatly warped.	Black - reddish brown (over-fired). Highly porous and crumbly. Slip turned green - yellow.
ZP18	A. 1. clearance	Small jar.	Black (over-fired). Many 'bubbles'. Surface dark purple-brown.

ZP19	A.10.3.20	Small jar.	Dark grey (over-fired). Sandy grits.
ZP20	A.10.8.39b	Open bowl.	Deep red. Wide dark grey core. Many small white grits.
ZP21	A.10.35.59	Very thick warped sherd.	Grading from black at the surface to reddish brown at the centre (over-fired). Many sandy grits. Heavily encrusted.
ZP22	A.11.3.5	Body sherd of a large vessel; melted into folds with surfaces fused together.	Black - deep wine red - brown (over-fired). Some 'bubbles'.

Table 2 Composition of the pottery samples. (Concentrations in ppm except for K, Na and Fe, which are in %).

	<i>ZP1</i>	<i>ZP2</i>	<i>ZP3</i>	<i>ZP4</i>	<i>ZP5</i>	<i>ZP6</i>	<i>ZP7</i>	<i>ZP8</i>
%K	1.43	1.21	1.99	1.86	1.59	1.31	1.73	1.84
%Na ¹	0.147	0.174	0.157	0.138	0.145	0.148	0.138	0.189
%Fe	3.23	3.54	4.75	4.07	3.41	3.43	3.53	4.26
Rb ¹	92.0	56.2	102.	99.6	50.2	50.7	54.8	84.0
Cs	2.91	5.18	3.64	3.47	2.28	1.64	2.63	3.65
Sc	13.6	18.7	20.3	20.8	14.4	15.0	14.9	21.7
La	21.6	46.3	26.1	24.0	22.0	18.7	20.3	23.3
Ce	47.5	114.	61.9	60.3	49.2	41.8	45.5	56.2
Eu ¹	0.935	2.42	1.10	1.13	1.61	1.27	1.21	1.54
Lu	0.374	0.895	0.456	0.498	0.322	0.274	0.285	0.365
Th	7.19	12.7	8.52	9.41	6.50	6.03	6.10	8.77
Cr	87.8	110.	130.	144.	83.9	83.4	86.7	142.
Co	15.5	11.2	24.1	17.6	14.6	18.1	15.3	18.4
Sm	4.07	12.6	5.65	5.22	5.15	4.19	4.67	5.41
	<i>ZP9</i>	<i>ZP10</i>	<i>ZP11</i>	<i>ZP12</i>	<i>ZP13</i>	<i>ZP14</i>	<i>ZP15</i>	<i>ZP16</i>
%K	1.83	1.84	2.06	2.14	2.02	2.41	1.98	2.01
%Na ¹	0.198	0.170	0.195	0.208	0.205	0.168	0.254	0.174
%Fe	4.29	4.51	5.04	4.31	5.26	5.32	5.14	4.61
Rb ¹	69.6	78.6	79.8	81.2	87.3	81.3	77.2	70.1
Cs	2.57	1.97	3.80	3.24	3.44	3.63	3.32	3.02
Sc	17.2	18.0	20.6	18.0	20.4	21.7	20.5	17.7
La	26.7	26.1	27.3	26.2	27.2	23.9	36.1	28.1
Ce	64.2	60.5	60.9	57.9	67.0	62.3	83.6	66.9
Eu ¹	1.73	1.62	1.76	1.61	1.54	0.992	2.00	1.75
Lu	0.387	0.377	0.388	0.370	0.406	0.353	0.442	0.440
Th	7.59	7.55	8.05	7.68	8.24	7.43	8.59	8.28
Cr	97.1	107.	120.	108.	111.	111.	114.	105.
Co	21.0	20.6	23.4	21.2	23.9	21.2	23.6	20.3
Sm	6.23	5.90	6.15	5.80	6.05	5.32	8.17	6.57

	<i>ZP17</i>	<i>ZP18</i>	<i>ZP19</i>	<i>ZP20</i>	<i>ZP21</i>	<i>ZP22</i>
%K	1.99	1.71	*	2.27	1.59	1.92
%Na ¹	0.238	0.152	0.136	0.251	0.218	0.120
%Fe	5.12	4.45	4.12	5.43	3.26	4.01
Rb ¹	79.0	78.2	65.0	81.0	58.1	60.7
Cs	3.54	3.36	2.81	4.27	2.71	2.77
Sc	20.2	20.7	18.8	23.2	13.7	16.3
La	29.9	27.8	23.0	32.3	23.2	26.0
Ce	68.7	63.0	53.9	61.2	53.7	61.0
Eu ¹	1.76	1.40	1.06	1.49	0.985	1.19
Lu	0.431	0.385	0.278	0.437	0.268	0.353
Th	8.77	9.02	7.54	10.7	7.87	9.09
Cr	120.	141.	114.	141.	80.6	98.0
Co	23.5	17.3	19.2	22.9	14.5	18.0
Sm	6.48	4.94	4.69	6.60	4.17	5.27

¹ Elements not considered in the statistical analyses.

* Missing value.

Table 3 Composition of the clay samples. (Concentrations in ppm except for K, Na and Fe, which are in %).

	<i>ZC1</i>	<i>ZC2</i>	<i>ZC3</i>	<i>ZC4</i>	<i>ZC5</i>	<i>ZC6</i>	<i>ZC7</i>
%K	*	*	*	*	1.16	1.05	0.565
%Na ¹	0.090	0.054	0.073	0.040	0.181	0.111	0.075
%Fe	2.13	1.59	1.74	0.821	2.63	2.34	1.30
Rb ¹	37.5	14.0	18.1	12.4	39.1	30.5	*
Cs	1.30	0.684	0.893	0.499	1.54	1.68	0.496
Sc	7.76	4.30	4.91	2.87	10.5	8.62	4.67
La	13.0	9.43	8.79	6.52	17.9	17.4	7.89
Ce	29.0	21.1	19.1	12.5	42.1	34.9	19.0
Eu ¹	0.694	0.451	0.432	0.280	1.08	1.07	0.347
Lu	0.161	0.096	0.115	0.086	0.248	0.283	0.101
Th	3.45	1.85	2.19	1.17	5.48	5.05	2.05
Cr	41.8	23.3	28.6	14.6	70.4	56.7	28.4
Co	7.11	5.77	3.26	2.53	11.3	9.37	4.54
Sm	2.70	1.96	1.74	1.30	3.48	3.49	1.59

¹ Elements not considered in the statistical analyses.

* Missing value.

Table 4 Classification of samples given by the k-means method at k=5.

<i>Cluster</i>	<i>Pottery</i>	<i>Clay</i>
1	ZP3,4,8-20,22	
2	ZP1,5-7,21	
3	ZP2	
4		ZC2-4,7
5		ZC1,5,6

Table 5 Composition of clusters 1 and 2. (Concentrations in ppm except for K, Na and Fe, which are in %).

	<i>Cluster 1</i>		<i>Cluster 2</i>		<i>Mean 2 /</i>
	<i>Mean</i>	<i>σ</i>	<i>Mean</i>	<i>σ</i>	<i>Mean 1</i>
%K	1.99	0.18	1.53	0.16	0.77
%Na ¹	0.185	0.040	0.159	0.033	0.86
%Fe	4.67	0.48	3.37	0.12	0.72
Rb ¹	79.7	10.9	61.2	17.5	0.77
Cs	3.28	0.55	2.43	0.50	0.74
Sc	19.8	1.9	14.3	0.6	0.72
La	27.1	3.4	21.2	1.7	0.78
Ce	63.1	6.7	47.6	4.4	0.75
Eu ¹	1.48	0.30	1.20	0.27	0.81
Lu	0.398	0.052	0.305	0.044	0.77
Th	8.45	0.86	6.74	0.78	0.79
Cr	119	16	84.5	2.88	0.71
Co	21.0	5.4	15.6	1.5	0.74
Sm	5.90	0.84	4.45	0.46	0.75

¹ Elements not considered in the statistical analyses.

Table 6 Final classification of the samples

<i>Cluster</i>	<i>Pottery</i>	<i>Clay</i>
A	ZP1,3-22	
B	ZP2	
C		ZC1,5,6
D		ZC2-4,7

Table 7 The composition groups. (Concentrations in ppm except for K, Na and Fe, which are in %).

	<i>Group A (21 spls)</i>			<i>Group B</i>	<i>Group C (3 spls)</i>		
	<i>Mean</i>	<i>σ</i>	<i>c. v.</i>	<i>(ZP2)</i>	<i>Mean</i>	<i>σ</i>	<i>c. v.</i>
%K	1.88	0.27	14.2	1.21	1.11	0.08	7.12
%Na ¹	0.179	0.039	22.2	0.174	0.127	0.048	37.5
%Fe	4.36	0.71	16.2	3.54	2.37	0.25	10.5
Rb ¹	75.3	14.7	19.5	56.2	35.7	4.6	12.8
Cs	3.08	0.64	20.8	5.18	1.51	0.19	12.7
Sc	18.5	2.9	15.6	18.7	8.95	1.38	15.4
La	25.7	4.0	15.5	46.3	16.1	2.7	16.8
Ce	59.4	9.1	15.4	114.	35.3	6.5	18.5
Eu ¹	1.41	0.31	22.1	2.42	0.948	0.220	23.2
Lu	0.376	0.064	17.0	0.895	0.231	0.063	27.3
Th	8.04	1.11	13.8	12.7	4.66	1.07	23.0
Cr	111.	20.	18.5	110.	56.3	14.3	25.4
Co	19.7	3.2	16.2	11.2	9.26	2.10	22.7
Sm	5.56	0.98	17.7	12.6	3.22	0.45	14.0

	<i>Group D (4 spls)</i>		
	<i>Mean</i>	<i>σ</i>	<i>c. v.</i>
%K ²	0.565	-	-
%Na ¹	0.0605	0.0168	27.8
%Fe	1.36	0.40	29.7
Rb ¹	14.9	2.95	19.8
Cs	0.643	0.188	29.2
Sc	4.19	0.91	21.8
La	8.16	1.26	15.4
Ce	17.9	3.7	20.8
Eu ¹	0.377	0.079	21.0
Lu	0.0994	0.0122	12.3
Th	1.81	0.45	24.7
Cr	23.7	6.6	27.7
Co	4.03	1.43	35.5
Sm	1.65	0.28	16.8

¹ Elements not considered in the statistical analyses.

² In group D, the K concentration is that for ZC7. The values for ZC2-4 are missing.

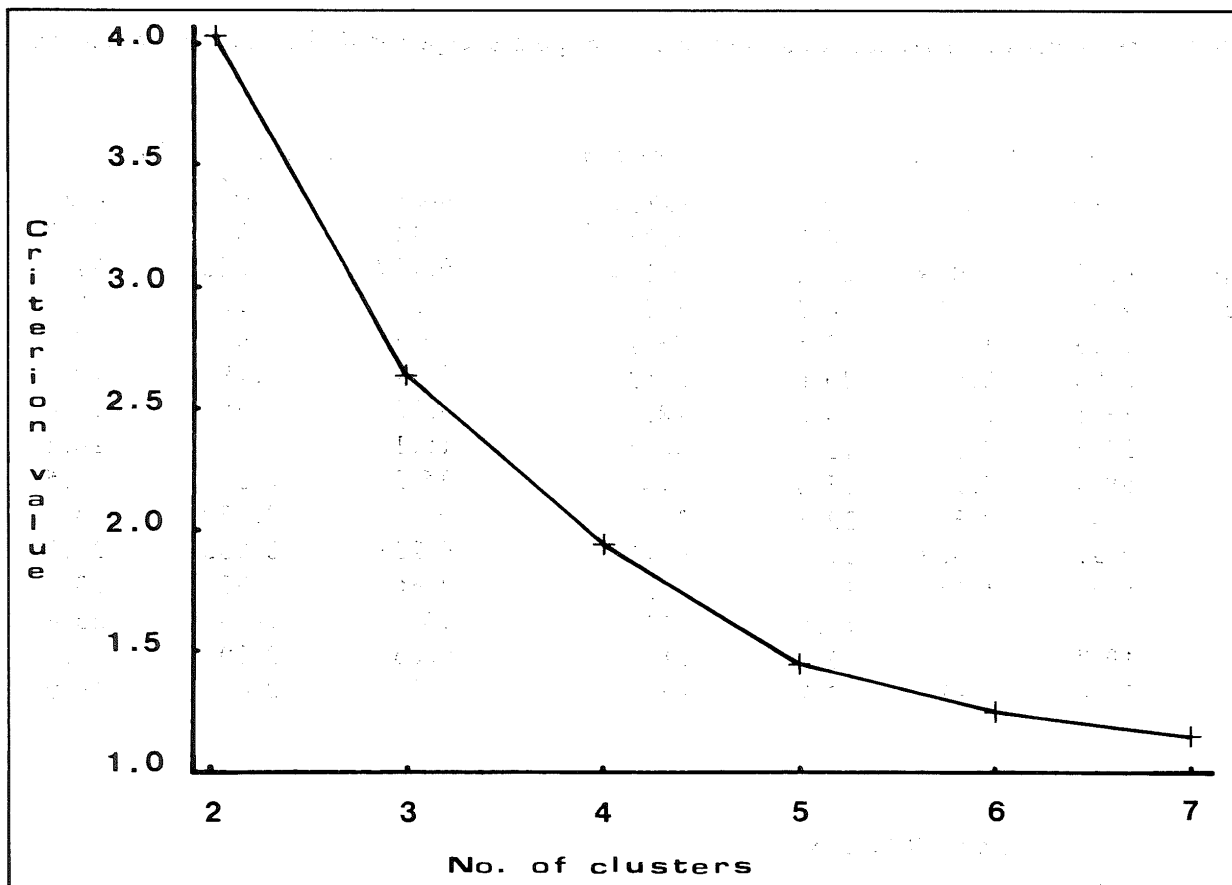


Fig. 1

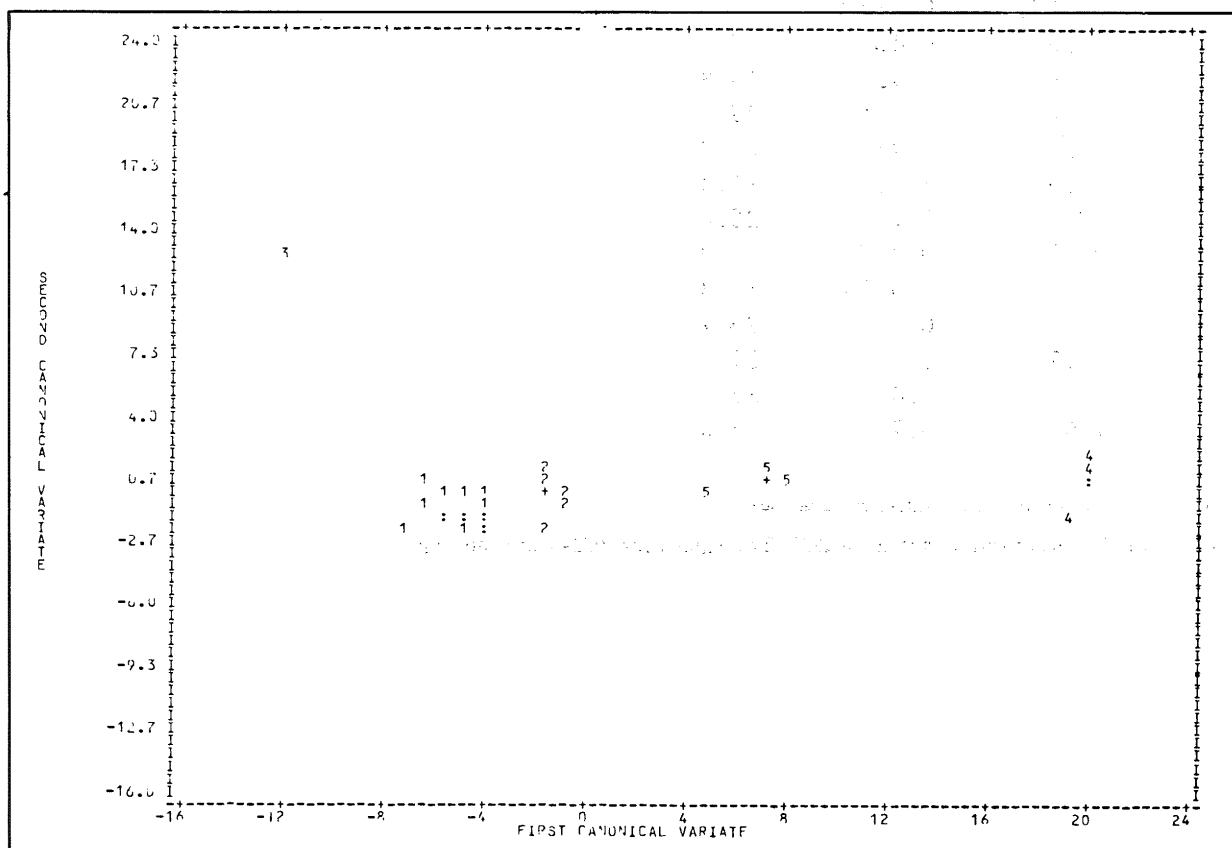


Fig. 2

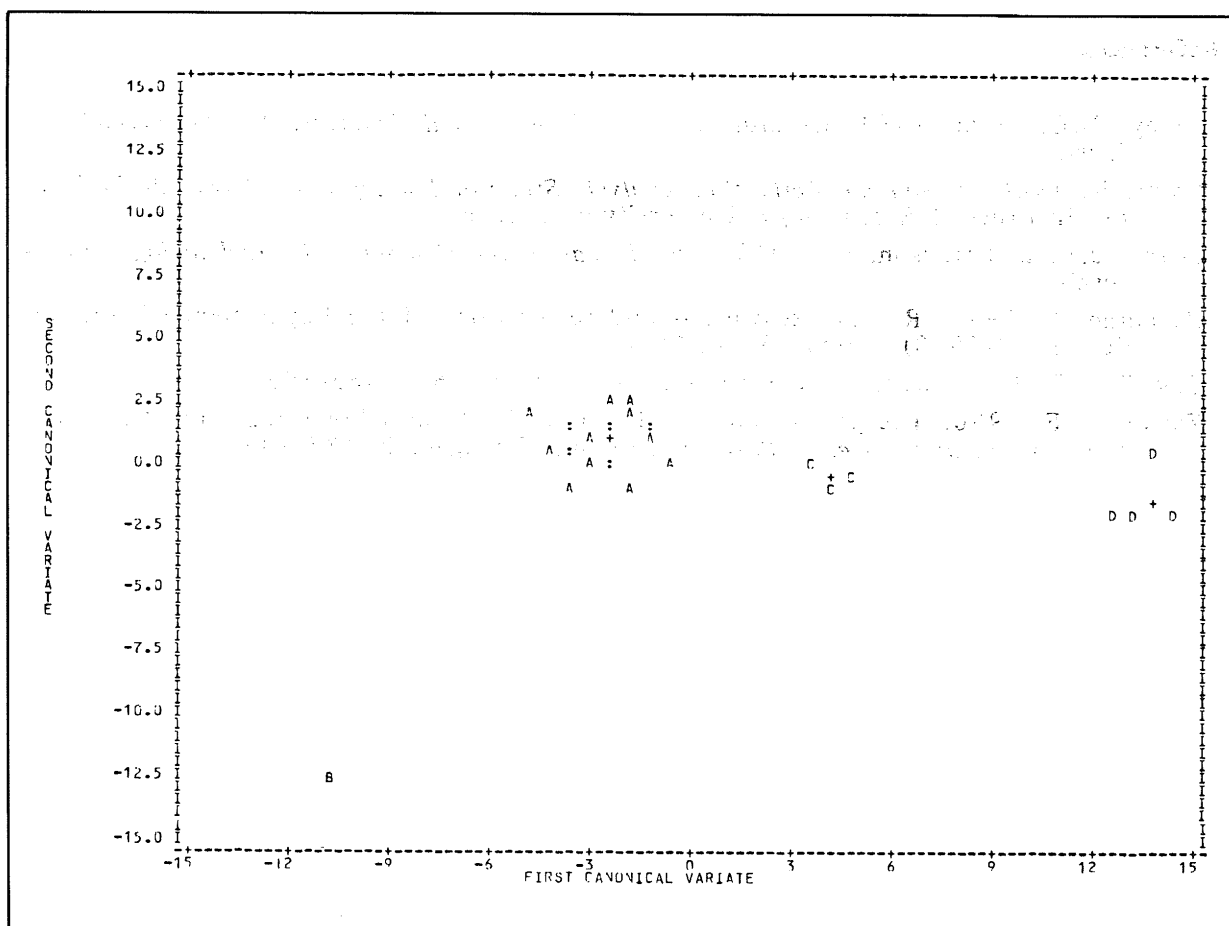


Fig. 3

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