

## POST-DEPOSITIONAL ELEMENTAL ALTERATIONS IN POTTERY: NEUTRON ACTIVATION ANALYSES OF SURFACE AND CORE SAMPLES\*

A. SCHWEDT, H. MOMMSEN and N. ZACHARIAS†

*Helmholtz-Institut für Strahlen- und Kernphysik, Universität Bonn, Nussallee 14–16, D-53115 Bonn, Germany*

*From 38 sherds with ‘corroded’ surfaces, two samples per sherd were examined using Neutron Activation Analysis, one from the surface and one from the core of each sherd, in order to analyse post-depositional alterations of minor and trace elements. The most striking effect was that a leaching of Ca could be found at the surface. Another group of elements that is severely affected are the alkali metals, with Cs and Rb showing the strongest changes. A third conspicuous group of elements are the Rare Earth Elements. Finally, the effect of such alterations on a statistical data evaluation to classify pottery according to provenance is investigated.*

**KEYWORDS:** POST-DEPOSITIONAL ALTERATION, POTTERY, NEUTRON ACTIVATION ANALYSIS, COMPOSITIONAL DATA, TRACE ELEMENTS, ALKALI METALS, RARE EARTH ELEMENTS, STATISTICAL GROUPING

### INTRODUCTION

The question of whether the chemical composition of ancient pottery is preserved during its burial in the soil is of major interest for the archaeometric field of provenancing, as knowledge about post-depositional alteration mechanisms may help to avoid misassignments when classifying chemical data. Such alteration processes have been investigated in the past, basically by three different approaches.

The most common approach has been that of comparative studies. Samples are analysed which, for archaeological reasons, are regarded as the products of a single workshop. If differences in the composition are observed, these are considered to be the results of alteration processes.

Examples of this approach are provided by, for example, Picon (1976, 1991), Franklin and Hancock (1980), Lemoine *et al.* (1981), Rottländer (1981, 1989), Buxeda (1999) and Buxeda *et al.* (2001). The dependency on the archaeological hypothesis, however, makes this approach risky—even if the assumption of a common origin is true, the potter might have changed his recipe for the production of the clay paste in the course of time, which would result in different ‘chemical fingerprints’ within the same workshop. Thus an observed deviation within the data sets need not necessarily be evidence for post-depositional alteration of the sherd.

Another approach has been made by simulation experiments. Laboratory test samples of known compositions are treated with different solutions under varying experimental conditions for up to several years. If, finally, the treated samples deviate in composition from the initial samples, these changes are also thought to be possible in natural alteration processes. Examples of this approach can be found in, for example, Hedges and McLellan (1976), Segebade and Lutz (1980) or Tubb *et al.* (1980). The uncertainty of this approach, however, is

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† Present address: Institute of Materials Science, N.C.S.R. Demokritos, Aghia Paraskevi, 15310 Attiki, Greece.

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the unanswered question of whether the conditions of a comparatively short-lived experiment can be compared to the actual situation of being buried for hundreds, or perhaps thousands, of years in natural soils.

An approach that avoids the problems mentioned above, and that is used in this paper, is that of profile or surface-core measurements. Several samples are taken from an ancient sherd that actually *was* buried in a natural soil for several thousand years. As the complete sherd can be assumed to have been produced from one well-homogenized paste, the initial compositions of all of the samples were identical. If the samples closer to the surface of the sherd now show a different composition compared to the samples from the core, this difference is direct evidence for alteration during the period of burial.

Besides these advantages, profile measurements also bear new possible risks. First, sherds may have been washed after the excavation, sometimes with slightly acidic solutions. According to Schneider *et al.* (1991, fig. 4a) and Schneider (private communication), however, this washing has a negligible effect on the sherd. On the other hand, the sherd might have been covered with a slip. Both problems can be avoided by removing the topmost part of the surface before the analysis.

Another possible cause of concentration gradients within a sherd could be a migration of elements within the sherd during the firing. However, a second series of profile measurements (to be published) also comprised sherds that had been fired at different temperatures. No gradient is evident in the measured elements, and so this possibility can be excluded.

In general, profile measurements have rarely been reported in the literature, and in most such cases only major elements have been measured. Examples of this approach can be found in, for example, Picon (1976, 1991), Dufournier (1979), Tubb *et al.* (1980), Lemoine *et al.* (1981) and Freestone *et al.* (1985).

#### SAMPLES AND ANALYTICAL TECHNIQUES

The idea behind this work was conceived during a sampling campaign in Greece, in 1995. Whenever a sherd was found to look corroded—that is, to have a soft or brittle surface—two samples were taken using a pointed corundum drill: one from a part close to the surface, after removal of the topmost part of the sherd with a corundum scraper (see above); and a second one from the core of the sherd, drilling at the same spot but deeper into the sherd.

A total of 38 sherds from 17 different sites were sampled in this way. Most of the sherds are fragments of Mycenaean vessels found on the Peloponnese (cf., Fig. 1). Additionally, one sherd from Rhodes, one from mainland Greece, one from Aegina and four from medieval or post-medieval sites in Germany have been included in the study.

All of the samples were analysed using Neutron Activation Analysis (NAA; for the experimental specifications, see Mommsen *et al.* 1991). Usually, both the surface sample and the core sample were irradiated in the same 'run' to avoid batch differences between possibly different irradiation conditions. The results are given in the Appendix. If enough powder was left after the NAA, X-ray diffractometry (XRD) was performed, using a Philips PW 1130 diffractometer and Cu-K $\alpha$  radiation ( $\lambda = 1.541838 \text{ \AA}$ ) in the range of 6–50° 2 $\theta$ .

#### DATA TREATMENT

It is evident that the selection of samples described above comprises products from many different workshops; in other words, the samples are members of many different chemical

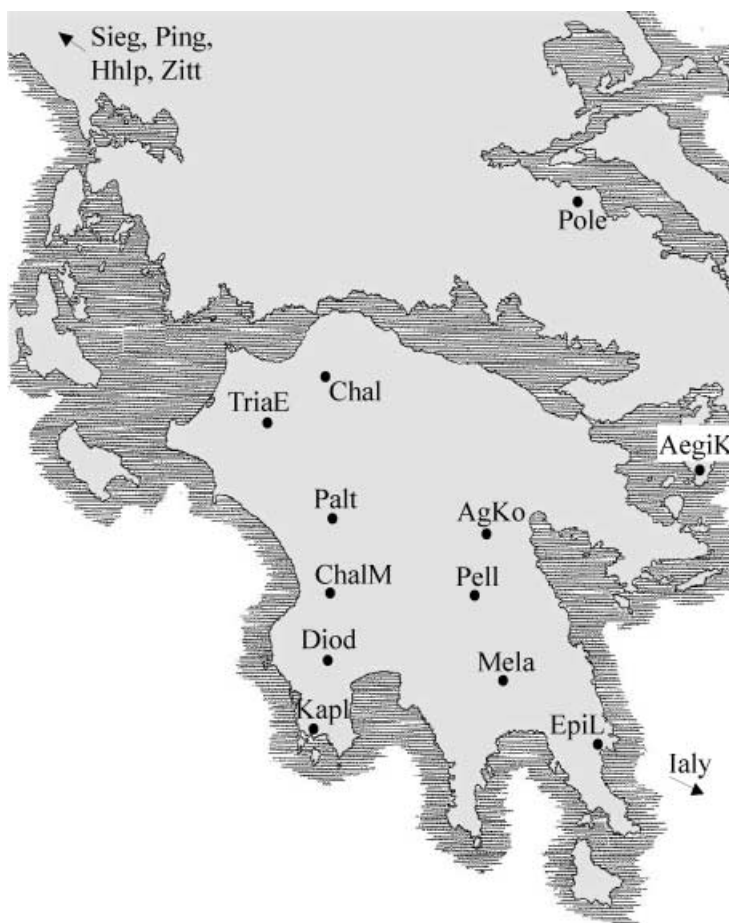


Figure 1 A map of the sites in Greece where the sampled sherds were found: Golemi (Pole), Chalandritsa (Chal), Agia Triada/Elis (TriaE), Palaeokastro (Palt), Agios Konstantinos (AgKo), Aegina Kolonna (AegiK), Pellana (Pell), Melathria Skoura (Mela), Epidavros Limera (EpiL), Chalkias/Messenia (ChalM), Diodia (Diod) and Kaplani (Kapl). Samples outside the area of the map have been found in Ialysos, Rhodes (Ialy), and in Germany at Siegburg (Sieg) and Pingsdorf (Ping), both in the Rhineland area, at Hohenleipisch, Brandenburg (Hhlp), and in Zittau, Saxony (Zitt).

groups. Thus the obtained concentrations  $c$  can only be used to compare the surface sample and the corresponding core sample. To go one step further, and to find trends of alteration within the complete data set, only the degree of alteration expressed as the change in the relative (weight-)concentration between the surface sample and the core sample,  $(c_{\text{Surface}} - c_{\text{Core}})/c_{\text{Core}}$ , can be used. Some examples of typical results of this comparison between the surface sample and the core sample are given in Fig. 2. The relative change is plotted for every element. The errors result from counting statistics via error propagation.

Ialy 95 (Fig. 2, top) represents the case in which no significant changes in the elemental pattern could be found between the surface sample and the core sample. Although Ni, Ti and Zr give values that are far from zero, their statistical error is so large that zero is no more distant

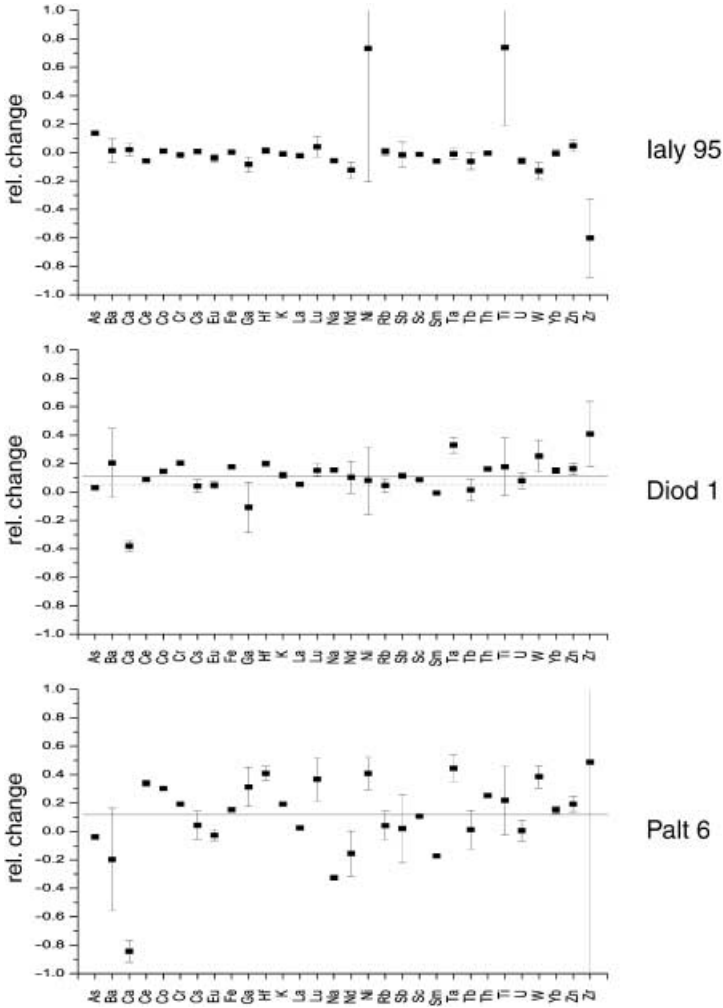


Figure 2 Examples of results from the comparison between the surface sample and the core sample (cf., text). Top, a sherd without changes (Ialy 95); centre, the leaching of a major element and its effect on trace element concentrations (Diod 1); bottom, a sherd with various changes (Palt 6).

than twice the statistical error. These three elements and Ca can be measured in Bonn only with large errors, because a change in the core of the Geesthacht reactor has recently reduced the flux of fast neutrons, which were necessary for interaction with these elements via nuclear reactions.

Another case is represented by Diod 1 (Fig. 2, centre). Here, nearly all of the measured elements show increased concentrations at the surface: only the concentration of Ca is about 38% lower in the surface sample (2.9%) than in the core sample (4.7%). The uniform increase of the other concentrations has been estimated over an average quotient of seven well-measured elements (Ce, La, Sc, Sm, Ta, Th and Yb). These are about 11% higher at the surface than in

the core sample (solid line). If we now assume that Ca was initially homogeneously distributed within the sherd, and that the lower concentration at the surface is the result of a leaching process, this would automatically result in a uniform increase of all of the other elements: the leaching of Ca reduces the total amount of material in the sample. Thus the percentage of an element with an unchanged absolute amount would be increased just by the reduction of the total amount (the 'constant sum problem'; cf., Rollinson 1993, 37ff.). If we further assume that the Ca is not leached only as Ca, but as  $\text{CaCO}_3$ , we can calculate the effect of this leaching on the other concentrations to be about 5% (dashed line). Thus, also considering the errors, the diagram for Diod 1 can be explained by leaching of calcite as the major process, while the other measured elements remain more or less unchanged.

The results for the vast majority of cases, however, can be represented by Palt 6 (Fig. 2, bottom). Again, the solid line marks the average increase, but the elements scatter to quite some extent around this value. In the surface sample, many elements have changed in various ways.

#### TRENDS IN THE WHOLE DATA SET

In order to find general trends, the behaviour of the whole data set was examined statistically. The results for elements with a special kind of behaviour are presented here.

#### *Ca*

The majority of the samples (29 out of 38) show lower Ca concentrations at the surface (cf., Fig. 3, left). As mentioned above, in recent times it has only been possible to measure Ca with a large statistical error. However, the data for the sherds with reduced Ca at the surface were recorded before the change in the reactor and so their statistical errors are small, whereas for the samples with higher Ca concentrations at the surface the errors are large. In the right-hand part of Fig. 3, the relative change divided by its error is plotted. The values of the relative change are, at a maximum, twice the statistical error above zero, but go down to 21 times the statistical error below zero. Thus, in spite of the Ca measurements with partly large errors, the decreased concentrations at the surfaces of the majority of the samples have to be considered as real.

Connected with a change in Ca is also one case in which enough powder was left to perform XRD on both the surface sample and the core sample. The diffractograms for samples Palt 30 and Palt 30s after background correction are shown in Fig. 4. In the core sample (black line) calcite ( $\text{CaCO}_3$ ) can be found, whereas in the surface sample (grey line) almost no calcite is left. This corresponds to the chemical data for Ca, with about 15% in the core sample and 4% in the surface sample. Thus, the chemical data show that Ca is leached out at the surface, and the diffractogram points to the calcite being the affected mineral. As the number of samples with reduced Ca values suggests, Ca—and especially calcite—tend to be affected by post-depositional alterations. However, not all of the samples with reduced Ca concentrations at the surface exhibit calcite peaks in the diffractogram, so calcite does not seem to be the only Ca mineral affected by leaching.

As mentioned above, changes in Ca or other major components of the sherd affect all other concentrations as well (as the short-lived isotopes of Si, Al and Mg cannot be measured using NAA in Bonn, we cannot say anything about these elements). Thus, before we look at minor or trace elements, the values of the relative change have to be recalculated to correct such indirect changes in the concentrations. Therefore, the concentrations of the surface sample are

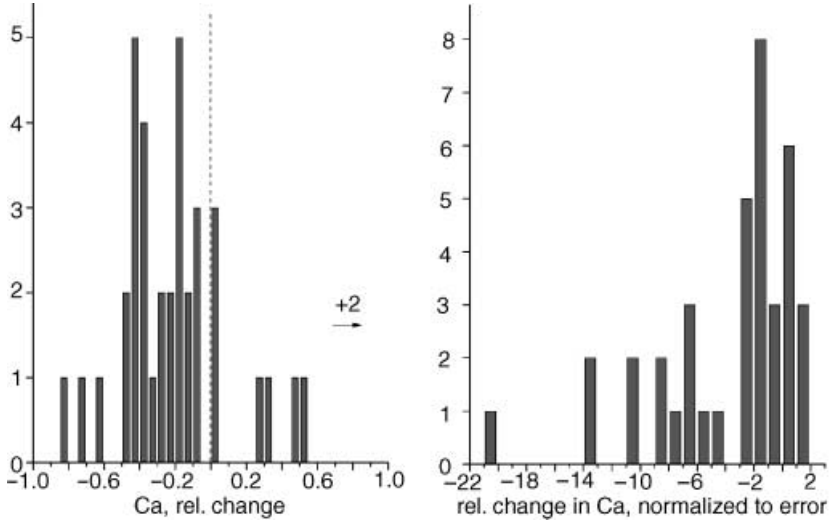


Figure 3 A histogram of the values of relative change for Ca, calculated from the raw data (left), and a histogram of the values of relative change normalized to their errors (right).

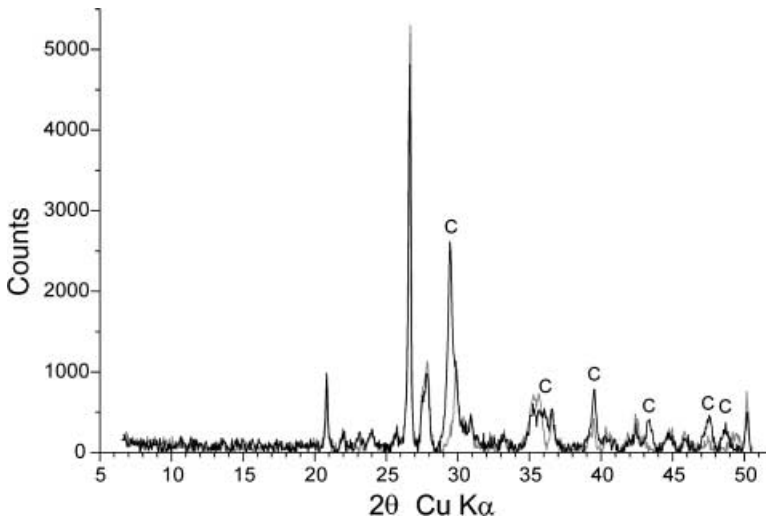


Figure 4 A comparison of the background-corrected diffractograms for samples Palt 30 and Palt 30s. The black diffractogram is that of the core sample and the grey one that of the surface sample. The positions of the calcite peaks are marked 'C'. It can clearly be seen that the calcite found in the core sample has disappeared in the surface sample.

fitted to that of the core sample by an average enrichment factor  $f$ , which is again calculated on the basis of Ce, La, Sc, Sm, Ta, Th and Yb. The recalculated relative change is then given by  $(f \cdot c_{\text{Surface}} - c_{\text{Core}}) / c_{\text{Core}}$ . The factor  $f$  shifts the baseline in diagrams such as Fig. 2 to the centre of the cloud of data points represented by the solid lines. Its use corresponds to comparing concentration ratios instead of the initial concentrations.

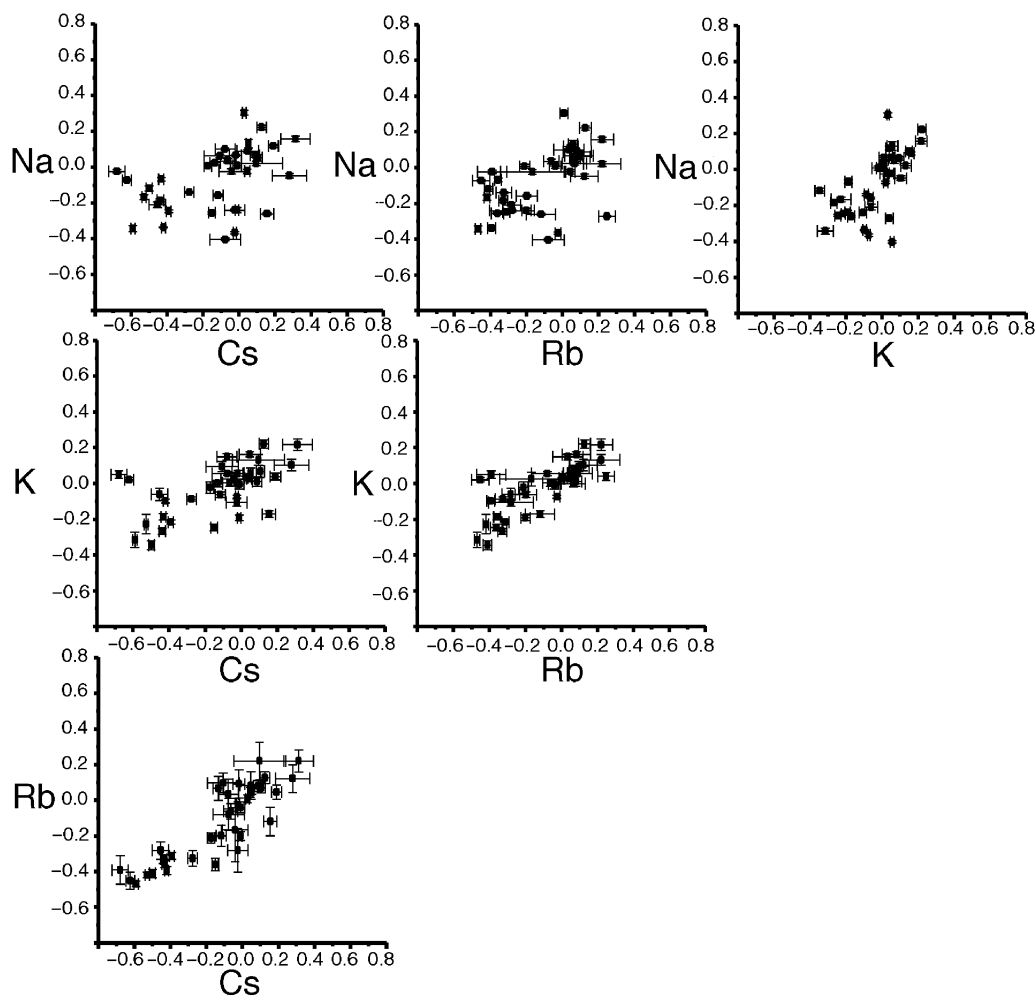


Figure 5 Changes in the measured alkali metals within the data set. All plotted data are the values of relative change after data correction.

### Alkali elements

The most striking differences between the surface and core samples could be found in the alkali metals. In Fig. 5, all possible correlation plots of alkali elements measured using NAA are given. In all of the diagrams, a major group of more or less unchanged samples can be observed, as can a second group with strongly reduced concentrations of at least one of the alkali metals at the surface. The strongest differences can be found for Cs in many samples. The concentration values of Cs are reduced by up to 68% at the surface. The change in Rb is correlated to that in Cs; however, for most of the samples the reduction in Rb is not as large as that in Cs. This might be explained in terms of differences in the ionic radius or in electro-negativity between the  $\text{Cs}^+$  and the  $\text{Rb}^+$  ions. The other alkali elements do not necessarily follow

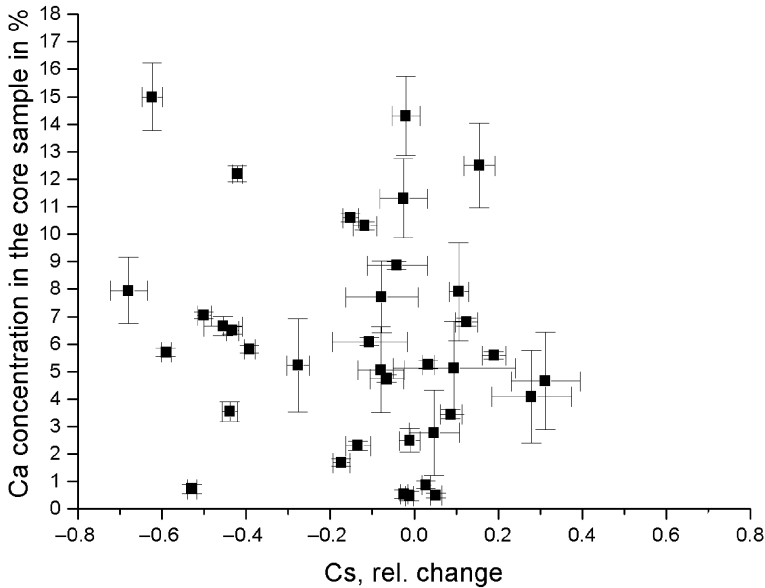


Figure 6 A plot of the relative changes in Cs between the core sample and the surface sample (after data correction) against the Ca concentration in the core sample.

Cs and Rb. In particular, as can be seen in the K–Cs and the Na–Cs diagrams, the samples with the strongest reduction in Cs show almost no change in Na and K. The changes in Na seem to be partly anticorrelated to changes in the other alkalines: if Na is reduced strongly, the other alkalines are reduced not so strongly in many of the changed samples, and vice versa. In total, 18 of the 38 samples are reduced in one or more of the alkali metals at the surface.

In order to consider other parameters that could have an influence on this behaviour, the first step was to look at the locations of the find sites of the samples. The altered samples were found at 9 of the 17 sites. If, on the other hand, we had more than one sample per site, usually not all of the samples were affected. Therefore, the behaviour is not specific for single sites.

In the past, alteration effects have mostly been reported for calcareous pottery. However, as Fig. 6 shows, the change in Cs can be found in non-calcareous samples as well as in samples with a very high Ca content.

As a final parameter, the firing temperature of the sherds was estimated, if possible, from the X-ray diffractograms. As in none of the cases was sufficient powder left to carry out refiring experiments, the estimation is only very rough. However, of the 11 samples with Cs and Rb reduced by over 20% at the surface, eight show quartz, plagioclase and pyroxene minerals having the most intense peaks in the diffractogram. Thus, according to the refiring experiments reported by Maggetti (1981) or Buxeda *et al.* (2002), a firing temperature of over 850°C can be assumed for these samples. This temperature makes a weathering process of a glassy phase possible, as already proposed by Picon (1976) for an observed leaching of K, or by Buxeda *et al.* (2002) for deviations in K and Rb.

On the contrary, among the samples with a Na reduction at the surface, some pieces exhibit intense peaks of calcite and illite muscovite, which suggests that a change in Na is also possible in low-fired ceramics.

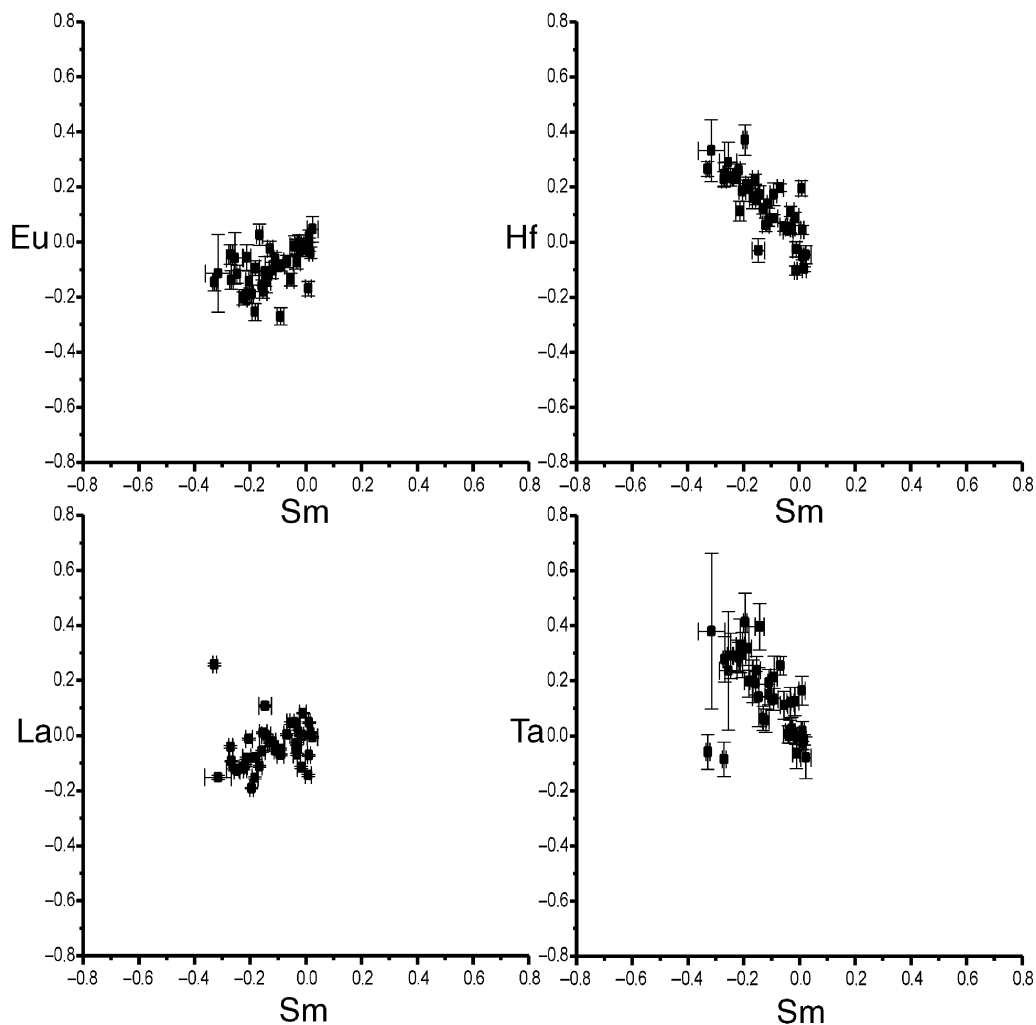


Figure 7 Relative changes in Eu, La, Sm, Hf and Ta (all data after data correction).

### Rare Earth Elements and others

Another kind of behaviour—not yet reported, to the best of our knowledge—can be found among the Rare Earth Elements (REE), in connection with Hf and Ta (cf., Fig. 7). In the Eu–Sm and La–Sm diagrams, the data points form much more compact clouds than in the case of the alkali metals; however, the ‘point of no change’ (0, 0) in both diagrams is found in the upper right-hand corner of this cloud, rather than in the centre. The relative changes of Sm, Eu and La are correlated to a certain degree.

The opposite effect can be seen in the Hf–Sm and Ta–Sm diagrams. Here, the relative changes are anticorrelated and the point (0, 0) is found in the lower right-hand corner of the clouds.

Among the other measured REE, Tb is more or less correlated with Sm, Eu and Ta, whereas Ce and Yb give values that scatter symmetrically around zero.

Whether these changes are as strong as indicated depends partly on the selection of elements used for the data correction that determines the position of the corrected baseline in diagrams such as Fig. 2 and, hence, the position of the (0,0) point. If the contributions of the REE are not considered in this correction, the observed changes between the surface and the core will be stronger for the REE, and smaller for Ta and Hf. On the contrary, if Ta is neglected, the new baseline will be closer to the values for the REE, resulting in larger deviations for Ta and Hf. In general, however, it can be seen that the values of the relative change are larger for Ta and Hf than for the REE. This means either that the REE are reduced at the surface (most strongly for Sm, Eu, La and Tb) or that Ta and Hf are enriched—or, most probably, both, since the correction also depends on the well-measured elements Sc, Th and Yb.

For the other elements measured, no clear tendencies or correlations could be found. The values of the relative change for As, Ba, Ce, Lu, Nd, Sb, Sc, U and Zr scatter more or less symmetrically around zero, whereas the distributions of some metals (Co, Cr, Fe, Th, W and Zn) tend to higher concentrations at the surface. The interpretation of this tendency, however, is uncertain, as it depends on the degree of data correction.

#### THE INFLUENCE ON THE STATISTICAL GROUPING

The grouping procedure in Bonn follows a multivariate filter method, based on a modified Mahalanobis distance as a similarity measure between the composition of a sherd and a given group (for details, see Beier and Mommsen 1994). Besides correlations, this distance considers the statistical errors of the individual samples and the spreads (the root of the mean-squared deviation) of the reference group, and is able to handle missing elemental values. In addition, possible dilutions or enrichments are considered by a best relative fit of the samples to the group mean values of the reference group, which corresponds to the use of concentration ratios during formation of the groups. A sample is considered as belonging to the reference group if its distance is smaller than a cut-off value, which is normally chosen to correspond to a 95% confidence level. Usually, in Bonn, 26 elements are considered during the statistical grouping (and also for the fit): these are Ce, Co, Cr, Cs, Eu, Fe, Ga, Hf, K, La, Lu, Nd, Ni, Rb, Sb, Sc, Sm, Ta, Tb, Th, Ti, U, Yb, W, Zn and Zr. The elements As, Ba, Ca and Na are normally neglected, as they are generally found to have high spreads within pottery groups: this can be due, for example, to different firing temperatures (As), different amount of temper (Ca, Na) or post-burial alterations (Ba, Ca), or they may already be inhomogeneous in the exploited clay bed.

In order to study the influence of the changes described above on the assignment to groups, a grouping was simulated taking the concentrations of the core samples as the reference group, with either the given statistical error or 5% as a minimal value for the spread. The data for the surface samples were filtered against these 'reference groups'.

In total, only 12 of the surface samples fulfil the normal grouping criterion (AegiK 43s, 55s and 91s; AgKo 10s; Chal 18s; Diod 1s; Hhlp 44s; Ialy 95s; Mela 2s and 7s; Ping 86s; and Zitt 19s). Two further samples would be assigned as 'associated' (ChalM 1s and Sieg 101s); in other words, two elements have to be neglected to fulfil the criterion. The remaining 24 would be classified as 'singles' or 'chemical loners', among which are the following:

- The 11 samples with strongly reduced Cs and Rb concentrations at the surface (Chal 11s; ChalM 7s; Epil 25s, 27s, 42s and 44s; Kapl 4s; Palt 23s and 30s; and Pell 5s and 14s). Cs and Rb also give the largest contributions to the distance in these cases.
- Another eight samples in which Sm and mostly also Eu and La give the highest contribution to the distance (AegiK 42s; ChalM 5s and 10s; Palt 6s, 28s and 46s; Pole 17s; and TriaE 6).

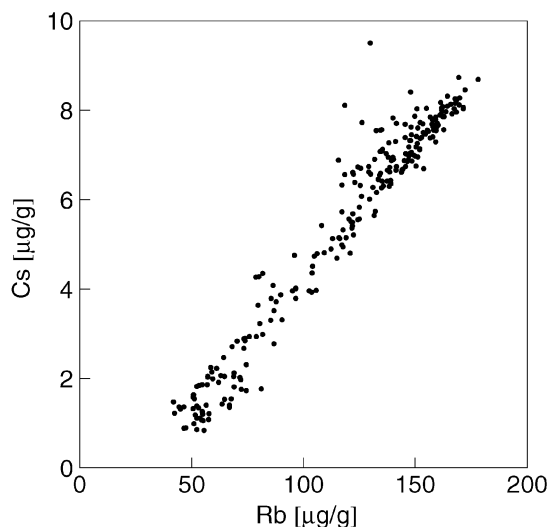


Figure 8 The Cs and Rb concentrations of 252 samples of pottery from Achaia and Elis. All other elements (with the exception of K) are very similar across all samples.

Ta and Hf, as well as the metals mentioned in the last paragraph (Co, Cr, Fe, Th, W, and Zn), only very seldom appear in the sequence of the most contributing elements. So the best relative fit over the usual 26 elements shifts the surface concentrations more towards the core concentrations of those elements, resulting in stronger deviations for the REE than with the reduced set of elements used for data correction in the preceding paragraphs.

- The remaining five surface samples: these show no regularity in the sequence of elements contributing to the distance.

As an example of possible alteration disturbing a real chemical classification, in Fig. 8 the Cs and Rb concentrations of 252 sherds of pottery from Achaia and Elis are plotted (Mommson *et al.*, to be published). Besides a main group, many samples have reduced concentrations in Cs and Rb (also K). All other elements are quite similar to the main group in these samples. During a normal statistical grouping, these samples would be subdivided into several groups according to the alkali concentrations. These groups would be thought of as representing different clay pastes, due to either a change in the procedure to refine the clay, or due to a change within the exploited clay bed. Given the results mentioned above, a possible explanation for this deviation could also be a post-depositional alteration process, which only affects the alkali elements.

#### CONCLUSIONS

It can be seen in this project that post-depositional alteration of ceramics, studied at corroded surfaces, may lead to changes in the compositional data that severely affect statistical grouping procedures. From the elements measured in Bonn, the ones most strongly affected by this alteration process are Ca and the alkali metals Cs, Rb, K and Na. All of them have been found to be strongly reduced in many of the surface samples.

Another group of elements that seem to be affected, as they show a deviating trend in comparison to the majority of elements, are the Rare Earth Elements, especially Sm, Eu and La.

However, it must be stressed that the samples described in this paper represent the pathological cases, that are and should be sorted out in any chemical classification of ancient pottery. Nevertheless, the observed effects might be one possible explanation as to why usually about 10% of singles occur in the statistical grouping of compositional data.

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APPENDIX

Table A A table of the raw data. Concentrations and average measurement errors are given in ppm (=  $\mu\text{g g}^{-1}$ ) unless indicated otherwise. 's' denotes the surface sample; '-' marks values missing due to experimental errors. Because of lack of space, only the average experimental errors are given

Sample	As	Ba	Ca (%)	Ce	Co	Cr	Cs	Eu	Fe (%)	Ga	Hf	K (%)	La	Lu	Na (%)
Aegik 42	9.61	328	11.3	44.6	22.2	336	5.01	0.87	4.07	7.82	3.92	1.88	20.3	0.32	0.89
Aegik 42s	23.3	654	5.84	61.8	24.0	494	6.28	1.05	5.83	9.50	6.50	2.16	22.9	0.53	0.87
Aegik 43	7.27	219	14.3	45.1	17.2	298	4.53	0.91	3.82	11.5	3.91	1.93	21.4	0.26	0.98
Aegik 43s	8.19	305	9.23	46.1	18.7	292	4.39	0.94	3.85	9.08	3.68	2.01	21.0	0.34	1.03
Aegik 55	10.9	319	12.5	48.3	23.7	378	5.89	0.97	4.28	15.0	4.11	1.90	21.9	0.34	1.03
Aegik 55s	26.1	236	4.96	75.3	31.5	507	9.62	1.23	6.15	20.1	5.64	2.23	34.3	0.53	1.08
Aegik 76	8.93	292	9.04	40.1	20.7	346	4.47	0.89	3.80	11.9	3.70	1.93	20.3	0.31	1.11
Aegik 76s	16.2	213	5.23	78.3	19.5	438	14.1	1.20	5.95	18.3	7.36	3.15	40.0	0.68	1.27
Aegik 79	7.92	282	10.6	38.5	15.7	350	4.93	0.82	3.41	15.6	3.33	2.02	19.7	0.30	1.11
Aegik 79s	19.4	330	5.30	86.6	19.8	414	6.71	1.25	5.69	13.4	6.56	2.44	30.3	0.75	1.32
Aegik 89	8.33	260	10.3	34.2	17.1	379	4.22	0.71	3.41	14.9	2.97	1.89	16.7	0.28	0.98
Aegik 89s	10.2	173	5.71	45.9	21.4	403	4.96	0.89	4.45	10.5	4.41	2.35	20.4	0.40	1.10
Aegik 91	6.36	366	8.86	40.7	15.9	305	4.95	0.78	3.43	17.6	3.37	1.89	19.1	0.30	0.86
Aegik 91s	12.7	904	5.79	56.2	20.9	373	6.07	0.88	4.82	21.8	5.76	2.48	20.7	0.38	1.08
AgKo 10	8.04	729	2.30	81.3	27.7	185	5.85	1.77	5.14	24.9	5.96	1.67	44.2	0.52	0.49
AgKo 10s	7.48	895	4.78	83.2	27.1	188	5.19	1.77	5.01	24.3	5.93	1.70	48.8	0.46	0.51
Chal 11	0.69	515	6.65	68.3	44.3	208	1.86	1.41	5.02	17.2	4.50	1.42	32.1	0.41	0.47
Chal 11s	7.64	658	5.41	65.3	45.3	279	1.00	1.16	5.30	23.3	5.31	1.32	27.1	0.52	0.37
Chal 18	6.75	544	2.49	71.2	26.8	255	4.81	1.10	5.91	31.7	4.71	2.14	30.5	0.53	0.92
Chal 18s	3.50	407	1.80	72.6	28.3	311	4.89	1.02	6.29	18.9	5.13	2.19	30.5	0.49	0.96
ChalM 1	7.32	313	5.13	74.8	32.7	225	0.75	1.46	5.79	22.2	4.67	0.95	37.4	0.49	0.30
ChalM 1s	7.35	178	5.22	74.6	35.3	298	0.90	1.57	6.91	19.7	5.61	1.19	36.3	0.53	0.34
ChalM 5	8.58	253	4.09	75.2	25.5	317	1.38	1.44	5.45	15.8	5.53	1.19	38.1	0.49	0.50
ChalM 5s	8.13	202	10.8	72.6	29.9	358	1.76	1.29	6.03	22.0	6.61	1.31	34.9	0.51	0.47
ChalM 7	6.06	278	3.55	73.5	41.8	390	4.80	1.59	5.52	25.0	6.51	1.78	38.2	0.55	0.45
ChalM 7s	4.46	177	3.00	73.8	45.7	426	2.76	1.41	6.24	28.0	7.04	1.34	40.9	0.51	0.38
ChalM 10	7.42	326	4.66	77.0	32.2	245	1.52	1.43	5.88	18.7	4.99	1.35	37.0	0.49	0.43
ChalM 10s	4.15	205	6.76	69.7	37.4	293	1.98	1.12	6.53	25.2	6.09	1.62	32.2	0.56	0.49
Diod 1	9.86	256	4.74	70.5	22.7	263	2.47	1.42	4.78	15.3	4.82	1.35	34.7	0.48	0.33
Diod 1s	10.2	308	2.94	76.6	26.0	316	2.58	1.49	5.62	13.6	5.78	1.51	36.6	0.56	0.38

Table A continued

Sample	As	Ba	Ca (%)	Ce	Co	Cr	Cs	Eu	Fe (%)	Ga	Hf	K (%)	La	Lu	Na (%)
Epil 25	13.1	427	7.05	61.4	23.9	208	4.39	1.21	5.08	14.2	4.11	1.84	30.4	0.41	0.48
Epil 25s	13.9	569	6.62	63.8	35.0	245	2.41	1.22	5.93	22.4	5.00	1.32	31.0	0.45	0.46
Epil 27	6.26	526	5.82	85.6	26.4	208	6.11	1.45	4.46	24.4	5.44	2.43	40.5	0.49	0.56
Epil 27s	5.74	409	4.46	91.7	33.2	261	4.01	1.43	5.30	26.6	6.69	2.05	42.1	0.57	0.46
Epil 42	14.1	535	6.50	77.3	22.5	228	5.46	1.49	4.61	14.6	6.01	2.16	41.5	0.48	0.52
Epil 42s	12.4	481	5.56	82.8	30.3	292	3.31	1.45	5.38	25.9	6.97	1.87	42.1	0.52	0.52
Epil 44	12.0	642	12.2	91.1	22.7	188	7.82	1.67	5.22	20.7	5.29	1.09	51.5	0.53	1.36
Epil 44s	16.2	700	10.5	98.3	32.1	214	4.89	1.84	5.82	25.5	5.95	1.06	51.5	0.57	0.97
Hhlp 44	7.05	507	0.50	56.9	5.99	71.6	10.3	0.75	1.32	23.4	6.93	1.76	29.5	0.36	0.19
Hhlp 44s	7.29	380	0.46	56.7	5.88	68.7	10.5	0.72	1.40	23.8	6.98	1.80	29.9	0.34	0.21
Ialy 95	8.53	469	5.25	78.6	19.6	122	10.8	1.21	5.59	30.9	4.62	2.08	37.4	0.44	0.19
Ialy 95s	9.70	476	5.36	78.0	19.8	120	10.8	1.16	5.60	28.4	4.69	2.06	36.5	0.46	0.18
Kapl 4	11.8	334	7.95	63.2	17.6	245	2.37	1.28	4.55	8.55	4.05	1.09	30.7	0.35	0.36
Kapl 4s	10.5	189	5.82	64.2	19.7	304	0.83	1.43	5.19	19.4	5.09	1.25	29.6	0.43	0.38
Mela 2	5.16	458	7.91	77.0	25.4	215	5.34	1.39	4.13	29.0	5.15	2.34	37.3	0.48	0.53
Mela 2s	5.36	422	4.58	72.4	25.9	213	5.60	1.30	4.15	31.1	5.15	2.36	34.3	0.46	0.52
Mela 7	4.43	318	3.45	90.5	21.8	196	5.34	1.60	4.11	16.6	6.20	2.35	43.7	0.49	0.84
Mela 7s	4.32	345	3.20	88.9	23.8	203	5.54	1.49	4.20	9.22	6.63	2.27	40.6	0.50	0.85
Palt 23	4.20	302	5.23	59.5	24.6	256	5.52	1.24	5.26	21.0	3.77	1.94	28.0	0.43	0.56
Palt 23s	3.64	337	3.00	56.8	25.8	306	4.06	0.92	5.44	20.0	4.51	1.80	26.5	0.39	0.49
Palt 28	10.4	332	6.80	59.0	29.6	226	5.33	1.15	4.84	13.6	3.22	1.76	29.6	0.41	0.47
Palt 28s	4.94	416	5.27	55.0	26.5	271	5.75	0.97	5.11	10.5	3.81	2.06	24.8	0.41	0.55
Palt 30	8.81	309	15.0	55.3	23.6	210	4.50	1.20	4.47	12.6	2.84	1.45	28.0	0.34	0.38
Palt 30s	7.08	465	3.84	53.4	29.3	305	1.82	1.05	5.70	22.1	4.19	1.59	24.3	0.41	0.38
Palt 42	8.99	300	1.69	67.4	17.5	161	4.50	1.26	4.15	12.0	5.54	1.30	33.1	0.44	0.51
Palt 42s	4.98	200	1.42	66.7	18.8	171	3.59	1.00	4.41	15.2	6.16	1.23	32.3	0.42	0.50
Palt 46	1.74	374	2.78	66.6	29.3	274	2.94	1.25	5.97	15.9	4.34	1.77	29.3	0.44	0.55
Palt 46s	2.91	444	3.66	57.5	30.4	330	2.72	0.95	6.13	19.0	4.55	1.82	25.6	0.44	0.53
Palt 54	4.30	332	5.06	57.1	27.9	282	2.72	1.01	5.44	19.7	4.96	1.77	23.6	0.44	0.75
Palt 54s	4.41	366	2.82	56.3	28.9	301	2.41	0.83	5.32	26.7	5.58	1.95	22.7	0.41	0.79
Palt 6	6.59	330	7.72	78.3	28.9	348	2.09	1.68	5.07	13.4	4.58	1.44	40.4	0.46	0.32
Palt 6s	6.34	265	1.21	105	37.7	415	2.18	1.63	5.85	17.6	6.45	1.72	41.4	0.63	0.22
Pell 5	8.35	572	5.71	93.8	33.2	194	7.08	1.69	5.14	19.1	5.53	2.50	47.3	0.50	0.48
Pell 5s	13.7	572	3.67	116	42.6	300	3.67	1.79	7.96	-	8.58	2.16	56.3	0.67	0.40

Pell 14	10.9	876	0.73	134	26.0	143	8.01	1.87	4.99	25.0	8.59	2.42	56.7	0.61	0.70
Pell 14s	8.54	556	0.59	127	31.3	191	4.06	1.86	6.41	22.2	11.0	2.00	61.1	0.71	0.63
Ping 86	16.8	485	0.88	89.5	5.24	146	16.9	1.47	1.67	27.9	5.13	1.85	44.5	0.38	0.13
Ping 86s	20.2	521	0.91	95.7	5.41	148	18.8	1.56	1.91	29.1	4.96	2.06	47.9	0.43	0.18
Pole 17	8.34	469	5.59	47.5	72.3	869	4.95	0.92	7.28	14.8	3.33	1.52	21.8	0.38	0.48
Pole 17s	7.39	417	3.44	40.9	83.7	1054	5.50	0.64	8.66	17.2	3.76	1.47	17.2	0.39	0.50
Sieg 101	2.06	681	0.48	149	7.30	177	15.0	1.70	1.49	39.9	8.10	1.13	81.4	0.46	0.21
Sieg 101s	0.98	666	0.61	176	7.69	199	17.2	1.94	1.85	49.1	8.87	1.06	98.3	0.54	0.19
TriAE 6	3.47	424	6.08	71.9	31.3	308	1.20	1.40	5.95	22.7	4.39	1.89	33.2	0.51	0.72
TriAE 6s	3.35	362	5.00	56.7	33.5	344	0.95	1.00	6.49	19.5	4.91	1.83	26.2	0.47	0.68
Zitt 19	4.36	615	0.54	122	5.23	87.4	20.4	1.65	1.05	41.5	9.13	2.64	62.7	0.61	0.16
Zitt 19s	9.06	651	0.83	146	6.99	98.7	23.4	1.86	1.20	45.6	9.65	2.87	74.2	0.72	0.12
Average measurement error	0.11	69	0.94	0.87	0.18	1.9	0.12	0.034	0.023	2.6	0.1	0.035	0.097	0.026	0.005
In %	1.3	16	20	1.1	0.7	0.7	1.8	2.6	0.5	12	1.8	1.9	0.3	5.5	0.8

Sample	Nd	Ni	Rb	Sb	Sc	Sm	Ta	Tb	Th	Ti (%)	U	W	Yb	Zn	Zr
Aegik 42	15.8	265	70.1	0.64	15.1	3.02	0.57	0.52	7.31	0.42	1.44	1.29	2.18	89.0	35.5
Aegik 42s	17.3	344	64.8	0.71	18.3	2.89	0.90	0.70	11.2	0.76	2.34	2.87	3.17	95.5	117
Aegik 43	14.4	268	62.1	0.57	14.8	2.91	0.64	0.63	7.30	0.69	1.22	1.21	2.15	82.7	35.3
Aegik 43s	20.1	241	66.9	0.58	14.8	2.94	0.58	0.71	7.30	0.48	1.52	1.53	2.15	84.3	34.8
Aegik 55	23.1	315	76.0	0.72	16.7	3.16	0.71	0.67	8.15	0.23	1.61	1.48	2.29	81.5	37.0
Aegik 55s	33.5	469	94.6	1.07	21.2	3.82	1.15	0.87	11.7	0.51	2.28	3.07	3.13	117	78.5
Aegik 76	18.5	299	67.4	0.54	14.3	3.36	0.59	0.52	6.51	0.30	1.81	1.35	2.11	104	183
Aegik 76s	18.3	260	132	1.99	22.5	3.54	1.31	1.06	13.3	0.62	2.92	2.76	4.54	228	214
Aegik 79	19.4	291	66.9	0.65	13.2	3.14	0.56	0.55	6.11	0.29	1.87	1.31	2.01	86.0	179
Aegik 79s	19.0	272	68.5	1.79	21.1	3.67	1.29	1.13	12.4	0.85	2.44	2.31	4.88	171	132
Aegik 89	13.6	348	58.6	0.47	12.7	2.88	0.48	0.53	5.52	0.30	1.64	1.14	1.75	72.8	141
Aegik 89s	19.3	331	62.5	0.75	15.4	3.02	0.84	0.67	8.46	0.73	1.82	1.33	2.54	110	49.2
Aegik 91	19.6	297	66.3	0.47	13.6	3.11	0.61	0.54	6.89	0.32	1.63	1.21	1.97	83.4	150
Aegik 91s	59.9	346	70.7	1.23	18.4	2.72	1.07	1.03	10.5	1.43	2.19	1.45	2.74	134	160
AgKo 10	31.8	224	95.2	1.21	20.7	6.25	1.19	1.20	14.3	0.66	2.15	2.51	3.70	128	41.4
AgKo 10s	32.1	209	104	0.94	20.7	6.32	1.14	1.23	14.1	0.56	2.48	2.53	4.03	152	47.3
Chal 11	29.1	262	56.9	0.60	18.5	5.74	0.90	0.89	11.4	0.38	1.99	2.12	3.22	112	231
Chal 11s	30.0	277	40.3	0.46	19.1	5.72	1.04	0.67	12.4	0.45	2.35	5.09	2.93	165	265

Table A. continued

Sample	Nd	Ni	Rb	Sb	Sc	Sm	Ta	Tb	Th	Ti (%)	U	W	Yb	Zn	Zr
Chal 18	31.6	231	109	0.51	20.3	5.40	1.02	0.80	13.3	0.50	2.37	2.67	2.91	115	248
Chal 18s	23.5	274	108	0.61	21.4	4.87	1.11	0.74	13.8	0.58	2.60	2.54	3.21	185	235
ChalM 1	31.5	241	25.3	0.59	22.1	6.56	1.00	0.95	12.9	0.69	2.15	1.92	3.35	130	221
ChalM 1s	29.1	298	34.0	0.76	25.2	7.10	1.23	0.95	15.7	0.95	1.96	2.32	3.72	110	230
ChalM 5	30.8	265	37.6	0.58	19.2	6.38	1.04	0.86	12.4	0.63	2.42	1.58	3.41	116	290
ChalM 5s	28.3	304	42.0	0.72	20.3	5.20	1.24	0.75	13.9	0.99	2.39	2.48	3.44	149	307
ChalM 7	29.8	325	92.3	0.93	19.5	7.08	0.98	0.96	12.0	0.40	2.04	2.37	3.73	123	293
ChalM 7s	36.6	370	63.4	1.04	18.7	6.85	1.12	0.85	13.8	0.42	2.03	2.21	3.47	147	309
ChalM 10	27.4	241	47.4	0.58	22.0	6.33	1.02	0.87	13.5	0.66	2.31	2.56	3.25	106	270
ChalM 10s	26.5	280	57.2	0.58	23.4	4.85	1.31	0.73	15.4	0.68	2.93	2.70	3.52	120	276
Diod 1	30.0	220	56.9	1.35	16.7	5.53	0.96	0.95	11.4	0.45	2.09	1.80	3.21	80.6	175
Diod 1s	33.1	238	59.5	1.51	18.1	5.50	1.28	0.96	13.3	0.52	2.25	2.26	3.69	93.8	246
Epil 25	27.3	175	86.2	0.44	19.1	4.54	0.79	0.70	10.9	0.54	2.38	2.43	2.70	83.2	180
Epil 25s	25.1	281	55.5	0.45	19.9	4.80	0.97	0.72	13.0	0.48	2.81	2.88	3.02	105	199
Epil 27	35.9	190	126	1.04	20.6	6.60	1.21	0.86	14.8	0.52	3.40	2.31	3.30	113	197
Epil 27s	39.4	231	93.5	1.08	20.3	6.33	1.50	0.88	17.3	0.59	3.96	2.54	3.76	138	173
Epil 42	32.6	181	119	0.82	19.0	5.96	1.12	0.92	12.9	0.45	3.60	2.24	3.26	79.2	166
Epil 42s	28.6	199	81.1	0.87	19.0	5.77	1.36	0.95	15.1	0.45	3.53	2.46	3.52	105	296
Epil 44	35.7	195	106	1.32	22.9	7.61	1.29	1.07	15.4	0.75	3.88	1.65	3.75	75.7	196
Epil 44s	36.0	228	69.2	1.04	24.8	8.30	1.41	1.13	17.8	0.42	4.25	1.76	3.97	90.6	187
Hhlp 44	20.9	29.1	120	0.97	11.2	3.56	1.57	0.58	9.75	0.42	3.07	3.31	2.71	56.3	271
Hhlp 44s	14.1	84.7	122	1.18	11.2	3.31	1.52	0.67	9.65	0.71	3.21	3.35	2.41	60.9	311
Ialy 95	29.6	76.8	173	0.88	19.4	5.38	1.21	0.74	14.8	0.61	3.72	2.47	2.95	86.3	129
Ialy 95s	25.9	133	174	0.87	19.2	5.05	1.19	0.66	14.7	0.59	3.54	2.15	2.93	90.5	51.4
Kapl 4	25.8	208	46.4	1.60	16.4	5.18	0.81	0.86	10.3	0.55	2.51	1.42	2.93	63.8	37.3
Kapl 4s	21.6	253	30.7	1.86	18.1	4.68	1.06	0.90	13.6	0.58	1.98	1.98	3.30	67.1	41.6
Mela 2	27.5	178	115	0.77	19.0	5.12	1.10	0.83	12.7	0.42	2.94	2.16	3.15	110	261
Mela 2s	25.4	201	117	1.00	18.5	4.70	1.06	0.74	12.5	0.64	3.23	2.19	2.97	105	254
Mela 7	40.1	123	114	0.87	21.3	7.18	1.29	0.90	13.7	0.53	2.78	2.74	3.43	108	242

Mela 7s	39.6	110	118	0.95	21.6	5.97	1.31	0.86	13.1	0.55	2.74	2.24	3.39	106	232
Palt 23	23.3	233	105	0.55	21.8	4.17	0.85	0.75	11.5	0.36	2.08	2.45	2.82	80.0	42.0
Palt 23s	21.5	294	71.8	0.50	22.6	3.85	1.05	0.56	12.6	0.75	2.83	2.55	2.82	81.4	40.9
Palt 28	25.4	221	105	0.61	19.4	4.85	0.68	0.69	10.0	0.38	2.23	2.05	2.56	92.4	157
Palt 28s	23.5	232	113	0.44	20.9	3.50	0.84	0.55	10.6	0.39	2.16	2.44	2.65	82.2	163
Palt 30	20.3	192	78.3	0.64	18.1	4.49	0.62	0.80	8.86	0.67	2.06	1.41	2.46	84.4	38.3
Palt 30s	20.1	311	46.2	0.73	21.9	3.89	0.95	0.55	11.4	0.87	2.73	2.00	2.71	86.7	41.4
Palt 42	29.2	112	75.8	0.90	15.7	4.78	0.90	0.84	11.7	0.41	1.93	2.43	3.13	67.6	221
Palt 42s	23.2	135	57.7	0.96	14.7	3.90	1.07	0.62	11.5	0.46	2.44	2.15	2.95	77.9	240
Palt 46	25.4	288	78.3	0.52	21.6	4.91	0.89	0.79	12.7	0.71	2.38	2.38	3.04	103	41.4
Palt 46s	20.2	312	75.0	0.50	21.5	3.46	1.02	0.60	10.1	0.57	2.19	2.43	2.88	104	42.0
Palt 54	21.1	202	67.8	0.58	20.9	3.72	0.87	0.75	11.2	0.61	1.90	1.89	2.76	94.5	40.8
Palt 54s	16.9	244	67.3	0.62	18.1	3.06	1.16	0.61	10.1	0.72	1.68	2.27	2.72	98.8	39.0
Palt 54s	36.4	265	66.2	0.69	23.4	7.15	0.99	1.12	12.9	0.63	2.91	2.22	3.81	98.7	42.2
Palt 6	30.7	374	68.9	0.70	25.9	5.92	1.43	1.13	16.2	0.76	2.93	3.07	4.39	118	62.8
Pell 5	45.3	193	149	1.22	22.1	7.54	1.30	1.02	15.3	0.45	3.26	2.14	3.49	119	233
Pell 5s	47.8	236	99.9	1.73	26.1	8.04	1.96	1.05	21.6	0.65	4.48	4.66	4.74	168	354
Pell 14	54.1	85.1	156	1.91	24.2	9.03	1.85	1.16	19.5	0.62	4.61	2.83	4.25	165	380
Pell 14s	58.5	117	97.3	2.20	20.5	9.03	2.49	1.18	24.9	0.85	4.81	4.77	5.13	409	447
Ping 86	37.1	31.8	122	1.52	17.9	6.67	1.31	0.87	14.1	0.49	3.89	2.29	2.79	75.6	221
Ping 86s	38.6	45.3	133	1.00	19.6	7.11	1.41	0.83	15.3	0.52	4.31	2.51	3.02	67.8	214
Pole 17	15.5	1133	83.8	0.51	21.8	4.02	0.71	0.50	8.17	0.36	1.32	1.93	2.25	111	128
Pole 17s	17.1	1459	81.8	0.50	22.0	3.06	0.87	0.46	8.63	0.54	1.48	2.14	2.23	132	108
Sieg 101	44.0	65.6	76.0	5.14	21.4	7.71	1.89	0.92	22.5	0.69	5.14	2.74	3.50	45.0	393
Sieg 101s	54.5	69.5	70.0	2.09	24.5	9.00	2.12	1.07	25.6	0.75	5.54	3.06	3.91	41.7	331
TriAE 6	32.4	252	55.0	0.41	22.2	5.55	0.98	0.76	12.7	0.47	1.82	2.16	3.19	108	115
TriAE 6s	22.4	316	53.5	0.50	23.6	3.86	1.12	0.53	12.9	0.53	1.98	2.34	2.88	141	152
Zitt 19	44.1	93.3	201	2.09	17.1	9.23	3.27	1.28	28.7	0.53	10.1	6.16	4.73	103	380
Zitt 19s	57.8	86.9	230	2.71	19.5	11.0	3.77	1.51	33.6	0.67	12.0	6.77	5.52	130	367
Average measurement error	3.8	25	3.4	0.088	0.031	0.053	0.048	0.065	0.1	0.079	0.19	0.17	0.062	3.1	34
In %	13	10	3.6	8.1	0.2	0.9	3.6	7.5	0.7	15	5.5	6.1	1.9	2.9	17