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The influence of different tempers on the composition of pottery

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ABSTRACT

The modification of the raw clay by the potter to produce a paste suitable for the intended purposes adds a layer of obfuscation to the problem of provenancing the original clay source by chemical fingerprinting. By preparing different pastes from the same commercially available raw clay and their chemical analysis by Instrumental Neutron Activation Analysis, an experiment under controlled conditions (tempers, mixing ratios, firing temperatures and sampling methods) sheds light on the influence of different tempers.

The results show that two different sampling procedures (drilling and grinding) have almost no influence on the chemical fingerprint with the exception of the elemental concentrations of As, Zr, and Hf. This may be due to the volatility of the compounds (As) or the presence of zircon crystals (containing Zr and Hf) which are partly lost during drilling. Three different firing temperatures show no significant influence as well.

The application of the modified Mahalanobis distance introduced by Beier and Mommsen in 1995 as a statistical filter and the introduction of a 'dilution factor' to the raw data show that the influence of quartz-dominated tempers can be filtered out of the data, resolving the underlying chemical fingerprint of the original clay source. At the same time, by mathematically removing the additional spread introduced by dilution, even subtle differences between similar pastes can be resolved by standard multivariate statistical means.

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1. Introduction

Archaeological artefacts can be identified and classified by means of scientific methods applied on this matter. This includes chemical analytical methods which aim at the establishment of a 'chemical fingerprint'. This term stands for a highly characteristic (trace) elemental composition of an archaeological artefact, allowing its provenancing by comparison with the chemical fingerprint of an object of proven origin.

This method has become a powerful tool in the identification and comparison of pottery with an archaeological background. Pottery consists mainly of clay – a material with a chemical fingerprint that often varies from one clay deposit to another, thus allowing its differentiation. Chemical and petrographic analyses have first been applied to pottery in the early 19th century by Wocel (see Pollard and Heron, 1996). A large amount of data on the chemical composition of pottery has been produced over the last decades (e.g. Jones, 1986; Perlman and Asaro, 1969; Alden et al.,

2006; Gliozzo et al., 2008; Tschegg et al., 2008). This enormous database, in theory, allows the grouping and identification of further finds of ceramic artefacts.

The potter usually modifies the original clay by levigation or tempering to clean the clay and prepare a material suitable for firing. Highly swellable clays need to be tempered with a non-swelling material to prevent cracking due to excess shrinkage during drying and firing. One problem that arises in the chemical analysis of pottery from this fact is that a clear identification of the clay source, potentially leading to a geographic localization, is thus usually impossible, as the clay is contaminated with foreign materials. However, the differentiation of recipes or prepared clay pastes is in many cases sufficient to solve the question of provenance. For tempers, many different materials have been used, for example organic material such as chaff or shell, grog (crushed ceramic), or various sands or crushed rocks.

Provenancing by means of the chemical fingerprint method is performed by comparison of the data of the unknown artefact with the database comprising the chemical data of objects with known origin. Comparison of data (in tabular or even in graphical form) can be problematic, especially with a large amount of data that have to be compared. How can one differentiate what a significant

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deviation is or what belongs still in the normal range of the natural fluctuations of the chemical fingerprint? To overcome this problem, several single- and multivariate statistical methods have been tried. However, since a separation of two different pastes is only possible if the error of the measurement is smaller than the differences between the two compositions, it is necessary to include the measurement errors into the statistical analysis. Furthermore it is possible that two batches of the same paste show small differences in their compositions since one batch was more 'diluted' than the other one (Perlman and Asaro, 1969). A dilution can occur when the potter adds more or less of the same temper to two different pastes. The addition of temper appears as a dilution of the original clay composition if the temper consists mainly of elements that are not investigated (i.e. quartz sand in case of Instrumental Neutron Activation Analysis, INAA). This dilution appears in the data as a uniform change in concentration and can be described mathematically as a constant factor, the 'dilution factor'.

To overcome the problems mentioned above, Beier and Mommsen (1994) proposed a modified Mahalanobis distance as a statistical similarity measure that incorporates the errors of the measurements as well as allows for the dilution factor mentioned above. This method has been applied successfully to many problems of provenancing (e.g. Hein et al., 2002; Mommsen et al., 1995; Schwedt et al., 2006).

The aim of the present paper is to experimentally show how different tempers will influence the composition of the finished sherd as well as how the modified Mahalanobis filter will react to those changed compositions. Also included in the experiment were some simple checks to see if and how different firing temperatures and different sampling techniques can influence the measured composition. A lot of studies have been published on the chemical analysis and provenancing of pottery (e.g. Abbott et al., 2008; Alden et al., 2006; Arnold et al., 2000; Demirci et al., 2004; Hall et al., 2002; Josephs, 2005; Mallory-Greenough et al., 1998; Neff and Bove, 1999; Perlman and Asaro, 1969; Pollard and Hatcher, 1994; Rathossi et al., 2004; Riederer, 2004; Schmitt, 1998; Tite, 2008). However, in most cases, ancient or contemporary pottery, produced under non-standardized conditions was used. By using commercially available clay to produce tempered and untempered samples which can be systematically analyzed without curatorial constraints, an experiment under strictly controlled conditions was possible, excluding any other sources of error.

2. Methods

2.1. Sample preparation

For the experimental study of the variation in the chemical composition of a given clay by the addition of different tempers, different firing temperatures as well as different sampling methods, a large quantity of (already levigated) clay was purchased from a local pottery. The dried clay contains approximately $55 \pm 10\%$ SiO₂, $27 \pm 2\%$ Al₂O₃, $4.4 \pm 0.2\%$ K₂O, $3.5 \pm 0.1\%$ FeO, and $0.26 \pm 0.04\%$ CaO, all measured by INAA. Aluminum values were not corrected for the ²⁸Si (n,p) ²⁸Al reaction, the crystal water content of the dried, unfired clay is approximately 7%.

For tempering, three different tempers were used: sand from the Sahara desert, commercial building sand and basaltic sand from Stromboli island, Italy. Each temper was investigated by optical microscopy using a Zeiss stereo-microscope STEMI V8 at a magnification of 16–128 \times as well as by INAA after washing three times with distilled water in an ultrasonic bath. In order to identify the mineral components in more detail, X-ray powder diffraction analysis (XRD) of the tempers was performed with a Siemens

D5000 instrument at the Natural History Museum in Vienna. The temper samples were ground in an agate mortar to a grain size of about 3 μ m and measured, using Cu K α -radiation over a 2 θ range from 2 to 65° (continuous scan, step size 0.01°, time step 2 s, synchronous rotation).

The sand from the Sahara desert (Grand Erg Occidental, near Ouargla, North Algeria) consists of, typically for aeolian transport, well rounded grains with an average diameter of 0.7 mm. Additionally a poorly rounded fine fraction of approximately 150 μ m diameter exists. XRD was carried out in addition to the inspection by optical microscopy. The results confirm that the grains are mostly quartz and some feldspars (albite). Zircon is present in form of typically idiomorphic prismatic bipyramidal crystals with a maximum length of about 150 μ m.

The building sand is of unknown origin and was collected from a local construction site. It consists of well sorted, angular grains of an average diameter of 400 μ m. The grains are mostly quartz with some feldspars (albite).

The basaltic sand from Stromboli was collected from the beach close to San Bartolo (Piscità). It consists of well sorted, angular grains with an average diameter of approximately 350 μ m. The grains are mostly black to greenish or gold-brown in color. A small fraction of the grains are bright red in color. The main components are volcanic slag fragments consisting of plagioclase, basaltic glass and, less frequently, augite and olivine.

The two quartz-dominated tempers were used since, due to its high availability and its firing properties, quartz was commonly used by the ancients as temper. The basaltic temper was chosen to give a good trace-element-rich counterpoint to quartz-dominated tempers.

For firing, four different pastes were prepared: one consisting of the original levigated clay and three pastes with one of the tempers added to the original clay, with a volume ratio of 120 ml clay to 40 ml temper, corresponding to an approximate wet weight ratio of 5:1. The paste was homogenized by intense manual kneading. From each paste, three tablets of about 8 cm diameter and a thickness of 0.8 cm were prepared, resulting in 15 tablets. The tablets were loosely covered with plastic film in order to ensure a constant drying rate to prevent cracking. After drying for three weeks, each of the three tablets from the same paste was fired at a different temperature, namely 900 °C, 1100 °C and 1200 °C; 1100 °C being the temperature used by the potter from whom the clay was purchased. Thus, for each paste one tablet was slightly underfired, one fired at the correct temperature and one slightly overfired. The firing was done according to the preferred method of the potter using a ROHDE TR305 burning kiln. The temperature was slowly raised (100 °C h⁻¹) to 600 °C and somewhat faster (250 °C h⁻¹) to the desired temperature. The final temperature was held for 20 min and the tablets were then kept in the closed kiln for slow cooling. The goal of this firing procedure was not to recreate the procedures used by the ancients but to yield experimentally sound samples, well fitted for our purposes. After firing, two samples were taken from each tablet, one by drilling with a fused alumina drill and one by breaking off a small piece that was ground in an agate mortar.

2.2. Instrumental Neutron Activation Analysis

The samples were measured according to the established procedure for geological samples in our institute (Steinhauser et al., 2006), as described below. All samples were dried for 12 h at 110 °C and weighed and sealed into Suprasil™ glass vials for irradiation in the central irradiation tube of the TRIGA Mk II reactor of the Atominstitut of the Austrian Universities at an approximate neutron flux density of $1 \times 10^{13} \text{ s}^{-1} \text{ cm}^{-2}$ for 40 h. After irradiation,

Table 1
Results of INAA for all samples without temper in $\mu\text{g g}^{-1}$ (1σ).

	T1G	T2G	T3G	T1D	T2D	T3D	ClayOrig
Na	7580 (91)	7430 (89)	7650 (92)	7560 (91)	7150 (86)	7550 (91)	7150 (86)
K	38,300 (728)	37,300 (597)	38,500 (847)	38,000 (950)	34,900 (1256)	38,300 (421)	36,300 (835)
Sc	23.27 (0.28)	22.83 (0.27)	23.20 (0.28)	23.70 (0.28)	21.63 (0.26)	23.44 (0.28)	21.76 (0.26)
Cr	154.6 (10.0)	150.1 (9.8)	149.1 (9.7)	158.3 (10.3)	144.1 (9.4)	155.8 (10.1)	142.5 (9.3)
Fe	29,400 (382)	28,800 (374)	29,200 (380)	29,800 (417)	27,300 (382)	29,600 (385)	27,400 (356)
Co	29.17 (1.20)	27.83 (1.14)	30.25 (1.24)	31.91 (1.31)	29.83 (1.22)	30.71 (1.26)	28.77 (1.18)
Zn	210.0 (3.4)	203.5 (3.3)	213.7 (3.4)	220.8 (3.8)	203.1 (3.7)	215.0 (3.4)	202.2 (3.2)
As	30.5 (1.3)	29.3 (1.2)	27.8 (1.2)	29.7 (1.2)	23.3 (1.0)	28.3 (1.2)	28.3 (1.2)
Rb	225.7 (7.7)	223.6 (7.6)	226.7 (7.7)	230.8 (8.1)	215.3 (7.5)	230.2 (7.8)	214.5 (7.3)
Zr	169 (13)	181 (20)	179 (13)	201 (29)	170 (16)	181 (24)	176 (19)
Sb	1.85 (0.12)	1.81 (0.10)	1.83 (0.11)	1.85 (0.10)	1.81 (0.14)	1.83 (0.10)	1.70 (0.10)
Cs	14.38 (0.17)	14.33 (0.16)	14.37 (0.16)	14.73 (0.18)	13.48 (0.13)	14.64 (0.16)	13.52 (0.15)
Ba	752.1 (37.6)	730.3 (26.3)	723.5 (34.0)	761.8 (39.6)	713.1 (27.1)	771.0 (37.8)	674.6 (39.1)
La	59.83 (0.90)	59.44 (0.89)	60.30 (0.90)	59.85 (0.90)	56.49 (0.85)	59.88 (0.90)	56.85 (0.85)
Ce	121.2 (3.0)	116.6 (2.9)	120.6 (2.9)	122.0 (3.1)	110.2 (3.2)	122.3 (3.1)	111.6 (2.8)
Nd	57.0 (10.3)	51.7 (2.3)	49.3 (2.3)	58.2 (3.2)	45.8 (4.7)	56.6 (9.3)	54.8 (8.3)
Sm	11.3 (0.3)	11.1 (0.3)	11.1 (0.3)	11.4 (0.3)	11.2 (0.3)	11.4 (0.3)	10.8 (0.3)
Eu	2.34 (0.06)	2.27 (0.06)	2.30 (0.06)	2.34 (0.07)	2.16 (0.06)	2.29 (0.06)	2.19 (0.06)
Tb	1.72 (0.10)	1.68 (0.10)	1.71 (0.10)	1.69 (0.10)	1.44 (0.08)	1.72 (0.10)	1.59 (0.09)
Yb	5.43 (0.31)	5.23 (0.24)	5.30 (0.07)	5.27 (0.07)	5.05 (0.10)	5.43 (0.07)	4.81 (0.07)
Lu	0.73 (0.06)	0.74 (0.07)	0.65 (0.05)	0.78 (0.12)	0.59 (0.16)	0.72 (0.02)	0.62 (0.02)
Hf	5.24 (0.09)	5.02 (0.08)	5.04 (0.09)	4.94 (0.04)	4.87 (0.06)	4.85 (0.09)	4.72 (0.08)
Ta	1.71 (0.05)	1.67 (0.05)	1.67 (0.05)	1.71 (0.05)	1.59 (0.04)	1.72 (0.05)	1.57 (0.04)
Th	20.4 (0.4)	20.0 (0.4)	20.1 (0.4)	20.7 (0.5)	19.1 (0.4)	20.6 (0.5)	18.9 (0.4)
U	4.93 (0.17)	5.04 (0.16)	5.14 (0.19)	5.24 (0.19)	5.00 (0.11)	5.23 (0.17)	4.95 (0.15)

the outer surface of the glass vials was decontaminated and the vials were packed into capsules fitting the automatic sample changer of the Atominstitut. The samples were measured for 1800 s after a decay time of 4 days and again after four weeks decay time for 10,000 s. The first measurement yielded the activities of the medium-lived radionuclides ^{24}Na , ^{42}K , ^{76}As , ^{140}La , ^{153}Sm and ^{239}Np (decay product of ^{239}U) and the second the activities of the long-lived radionuclides ^{46}Sc , ^{51}Cr , ^{59}Fe , ^{60}Co , ^{65}Zn , ^{86}Rb , ^{95}Zr , ^{124}Sb , ^{134}Cs , ^{131}Ba , ^{141}Ce , ^{147}Nd , ^{152}Eu , ^{160}Tb , ^{169}Yb , ^{177}Lu , ^{181}Hf , ^{182}Ta , and ^{233}Pa (decay product of ^{233}Th). All measurements were performed on a HPGe-detector (1.78 keV resolution at the 1332 keV ^{60}Co peak; 49% relative efficiency), connected to a PC-based multi-channel analyzer with a preloaded filter and a Loss-Free Counting system.

Together with the samples, internationally certified reference materials CANMET Reference Soil SO-1, MC Rhyolite GBW 07113, NIST SRM 1633b Coal Fly Ash, BCR No. 142 light sandy soil and NIST SRM 2702 Inorganics in Marine Sediment; and three samples of the Bonn Standard (Mommensen and Sjöberg, 2007) were irradiated and measured.

2.3. Statistical analysis

To statistically analyze the chemical similarity of the samples, a multivariate statistical filter method developed in Bonn (Beier and Mommensen, 1994) was applied. This statistical filter was developed explicitly for use in the provenancing of pottery. It

Table 2
Results of INAA for all samples with Sahara sand temper in $\mu\text{g g}^{-1}$ (1σ).

	S1G	S2G	S3G	S1D	S2D	S3D	Sahara sand
Na	5960 (72)	6430 (77)	6060 (73)	5600 (67)	5670 (68)	6250 (75)	2960 (36)
K	28,400 (483)	31,000 (496)	28,800 (490)	26,400 (581)	26,300 (763)	28,400 (653)	9000 (369)
Sc	16.04 (0.19)	17.15 (0.21)	16.44 (0.20)	14.65 (0.18)	15.25 (0.18)	16.08 (0.19)	1.34 (0.02)
Cr	105.4 (6.9)	113.6 (7.4)	107.5 (7.0)	95.6 (6.2)	100.9 (6.6)	106.5 (6.9)	7.5 (0.5)
Fe	20,600 (268)	22,000 (286)	21,200 (297)	18,800 (263)	19,500 (273)	20,700 (290)	2900 (44)
Co	19.18 (0.79)	22.24 (0.91)	21.74 (0.89)	18.80 (0.77)	20.64 (0.85)	21.49 (0.88)	0.76 (0.03)
Zn	141.5 (2.4)	157.0 (2.5)	152.2 (2.4)	134.4 (2.3)	145.7 (2.5)	150.4 (2.6)	0.0 (0.0)
As	21.2 (0.9)	20.4 (0.8)	18.6 (0.8)	19.5 (0.8)	17.1 (0.7)	18.3 (0.8)	0.4 (0.04)
Rb	170.6 (5.8)	173.9 (5.9)	167.5 (5.7)	156.5 (5.5)	161.6 (5.7)	165.6 (5.8)	33.8 (1.2)
Zr	192 (21)	201 (19)	202 (14)	160 (18)	162 (12)	196 (21)	250 (17)
Sb	1.29 (0.08)	1.32 (0.07)	1.29 (0.08)	1.21 (0.08)	1.27 (0.09)	1.25 (0.08)	0.06 (0.01)
Cs	10.25 (0.12)	10.62 (0.13)	10.18 (0.12)	9.48 (0.11)	9.72 (0.13)	9.94 (0.12)	0.50 (0.01)
Ba	600.9 (30.6)	627.9 (28.9)	613.7 (28.8)	561.8 (29.2)	625.0 (34.4)	594.6 (30.3)	295.3 (10.0)
La	42.43 (0.64)	45.94 (0.69)	43.91 (0.66)	42.02 (0.63)	44.93 (0.67)	42.78 (0.64)	9.21 (0.15)
Ce	87.5 (2.2)	91.1 (2.3)	91.0 (2.3)	83.6 (2.1)	91.7 (2.3)	84.7 (2.1)	18.9 (0.5)
Nd	45.9 (6.6)	41.4 (1.9)	41.1 (5.6)	38.2 (6.9)	37.6 (2.5)	37.3 (2.1)	8.8 (2.1)
Sm	8.3 (0.2)	8.6 (0.3)	8.1 (0.2)	8.3 (0.2)	8.6 (0.3)	8.1 (0.2)	1.5 (0.05)
Eu	1.65 (0.04)	1.79 (0.05)	1.69 (0.05)	1.52 (0.04)	1.59 (0.05)	1.68 (0.05)	0.35 (0.01)
Tb	1.25 (0.07)	1.29 (0.07)	1.25 (0.07)	1.15 (0.07)	1.22 (0.07)	1.22 (0.07)	0.18 (0.01)
Yb	3.91 (0.05)	4.05 (0.05)	3.78 (0.05)	3.77 (0.12)	3.84 (0.06)	3.92 (0.05)	0.88 (0.02)
Lu	0.56 (0.24)	0.58 (0.01)	0.54 (0.05)	0.49 (0.05)	0.51 (0.07)	0.52 (0.02)	0.16 (0.01)
Hf	6.06 (0.08)	6.16 (0.08)	6.23 (0.07)	5.10 (0.07)	4.75 (0.04)	5.66 (0.07)	8.79 (0.08)
Ta	1.30 (0.04)	1.30 (0.03)	1.24 (0.03)	1.15 (0.03)	1.34 (0.04)	1.21 (0.03)	0.48 (0.01)
Th	15.2 (0.3)	15.8 (0.3)	15.5 (0.3)	16.8 (0.4)	17.1 (0.4)	14.5 (0.3)	4.2 (0.1)
U	3.77 (0.13)	3.88 (0.13)	3.71 (0.13)	3.59 (0.13)	3.57 (0.16)	3.74 (0.13)	0.98 (0.07)

Table 3
Results of INAA for all samples with basaltic temper in $\mu\text{g g}^{-1}$ (1σ).

	B1G	B2G	B3G	B1D	B2D	B3D	Basaltic temper
Na	11,170 (134)	11,140 (134)	11,210 (135)	10,580 (127)	9860 (118)	9780 (117)	20,000 (240)
K	32,800 (590)	33,000 (594)	34,600 (623)	31,800 (763)	27,900 (837)	31,300 (783)	21,100 (907)
Sc	25.84 (0.31)	25.91 (0.31)	24.58 (0.29)	23.49 (0.28)	21.97 (0.26)	25.51 (0.31)	28.54 (0.34)
Cr	130.1 (8.5)	136.9 (8.9)	131.6 (8.6)	124.7 (8.1)	112.2 (7.3)	130.7 (8.5)	75.3 (4.9)
Fe	37,500 (488)	39,000 (507)	36,600 (476)	37,900 (493)	36,800 (478)	36,000 (468)	56,500 (735)
Co	28.38 (1.16)	32.69 (1.34)	27.79 (1.14)	31.48 (1.29)	30.81 (1.26)	30.88 (1.27)	28.35 (1.16)
Zn	166.5 (2.8)	179.8 (2.9)	163.1 (2.6)	176.1 (3.0)	160.5 (2.7)	176.2 (3.0)	0.0 (0.0)
As	22.4 (0.9)	22.2 (0.9)	22.0 (0.9)	22.2 (0.9)	19.8 (0.8)	19.5 (0.8)	5.3 (0.3)
Rb	178.0 (6.1)	184.3 (6.3)	178.0 (6.1)	189.0 (6.4)	164.8 (5.6)	178.5 (6.2)	72.4 (2.5)
Zr	167 (19)	184 (13)	162 (12)	185 (14)	144 (12)	142 (11)	189 (21)
Sb	1.32 (0.09)	1.37 (0.07)	1.33 (0.07)	1.39 (0.09)	1.25 (0.07)	1.32 (0.07)	0.24 (0.02)
Cs	11.49 (0.14)	11.76 (0.14)	11.32 (0.14)	11.99 (0.16)	10.47 (0.23)	11.42 (0.15)	4.81 (0.09)
Ba	799.0 (36.8)	824.8 (29.7)	757.9 (34.1)	886.8 (42.6)	777.1 (27.2)	773.6 (37.9)	1014.8 (43.6)
La	57.99 (0.87)	58.16 (0.87)	59.62 (0.89)	55.38 (0.83)	50.92 (0.76)	55.15 (0.83)	52.74 (0.79)
Ce	111.7 (2.8)	117.7 (2.9)	107.8 (2.7)	116.3 (2.9)	103.0 (2.7)	111.7 (2.8)	92.7 (2.3)
Nd	51.1 (2.4)	52.6 (2.3)	47.9 (6.1)	53.1 (2.8)	52.2 (11.3)	44.4 (2.6)	43.9 (2.5)
Sm	10.5 (0.3)	10.4 (0.3)	10.5 (0.3)	10.5 (0.3)	9.7 (0.3)	10.2 (0.3)	8.7 (0.3)
Eu	2.28 (0.06)	2.32 (0.06)	2.25 (0.06)	2.24 (0.06)	2.06 (0.06)	2.23 (0.06)	2.23 (0.06)
Tb	1.54 (0.09)	1.59 (0.09)	1.49 (0.09)	1.57 (0.09)	1.38 (0.08)	1.53 (0.09)	1.13 (0.07)
Yb	4.38 (0.06)	4.40 (0.06)	4.48 (0.22)	4.58 (0.25)	3.97 (0.07)	4.28 (0.06)	2.39 (0.05)
Lu	0.63 (0.02)	0.61 (0.05)	0.67 (0.08)	0.59 (0.08)	0.60 (0.02)	0.62 (0.08)	0.23 (0.01)
Hf	4.75 (0.09)	4.83 (0.08)	4.52 (0.09)	4.80 (0.10)	4.13 (0.04)	4.57 (0.09)	3.78 (0.08)
Ta	1.48 (0.04)	1.54 (0.04)	1.47 (0.04)	1.56 (0.04)	1.39 (0.04)	1.47 (0.04)	1.12 (0.03)
Th	18.9 (0.4)	19.7 (0.4)	18.3 (0.4)	19.7 (0.4)	17.6 (0.4)	18.6 (0.4)	16.2 (0.4)
U	4.74 (0.17)	4.81 (0.18)	4.77 (0.17)	4.96 (0.17)	4.44 (0.18)	4.78 (0.15)	4.14 (0.17)

considers the measurement errors for each individual concentration value as well as the spread of the groups formed.

$$d_{\text{mod}}^2(\vec{x}, \vec{y}) = \frac{1}{m-1} (f_0 \vec{x} - \vec{y})^T (f_0^2 S_x + S_y) (f_0 \vec{x} - \vec{y}) \quad (1)$$

This statistical filter consists of the modified Mahalanobis distance, as seen in Eq. (1). The measurement errors (S_x) of the sample and the spread of the group (S_y) are used for scaling. Additionally, the normalization to the number of elements measured ($1/(m-1)$) makes it possible to compare a sample where the detection limits were not exceeded for all elements. Furthermore, the filter has the ability to correct for uniform concentration changes as they occur due to the tempering (or levigating) of the clay during the

preparation of the paste. This dilution (already mentioned by Perlman and Asaro (1971)) is considered in the factor f_0 in Eq. (1). The dilution factor for a single element k can be calculated by the division of the two corresponding elemental concentrations $f_k = y_k/x_k$. The dilution factor for a sample \vec{x} with respect to another sample or group mean \vec{y} can thus be calculated from the mean of all single element dilution factors (see best relative fit, Harbottle, 1976). In this case however, since it is assumed that the dilution factor should bring two samples as close together as possible, it is in fact calculated as the solution of the partial derivative of Eq. (1) with respect to f_0 .

This statistical filter should be able to group pastes together if the tempers used in the production of the paste do not significantly

Table 4
Results of INAA for all samples with building sand temper in $\mu\text{g g}^{-1}$ (1σ).

	Bu1G	Bu2G	Bu3G	Bu1D	Bu2D	Bu3D	Building sand
Na	7190 (86)	7230 (87)	7030 (84)	7290 (87)	6790 (81)	7140 (86)	7160 (86)
K	33,000 (594)	32,300 (549)	31,200 (468)	30,100 (692)	30,300 (909)	32,800 (656)	14,000 (476)
Sc	18.02 (0.22)	17.56 (0.21)	16.47 (0.20)	15.88 (0.19)	16.98 (0.20)	18.39 (0.22)	1.05 (0.01)
Cr	117.9 (7.7)	114.6 (7.4)	105.9 (6.9)	103.4 (6.7)	111.8 (7.3)	120.5 (7.8)	5.2 (0.3)
Fe	22,900 (298)	22,300 (290)	20,900 (272)	20,200 (283)	21,600 (302)	23,400 (328)	1900 (30)
Co	21.85 (0.90)	21.91 (0.90)	20.09 (0.82)	21.47 (0.88)	24.12 (0.99)	24.90 (1.02)	0.55 (0.03)
Zn	160.6 (2.7)	158.9 (2.5)	148.8 (2.4)	148.0 (2.5)	165.8 (3.0)	172.4 (2.8)	6.1 (0.2)
As	23.9 (1.0)	20.9 (0.9)	20.7 (0.8)	21.2 (0.9)	27.9 (1.2)	19.5 (0.8)	0.9 (0.04)
Rb	185.0 (6.3)	181.4 (6.2)	171.3 (5.8)	168.1 (5.9)	173.3 (6.1)	187.6 (6.4)	49.4 (1.7)
Zr	140 (17)	154 (18)	135 (10)	144 (19)	143 (28)	157 (12)	32 (2)
Sb	1.57 (0.09)	1.44 (0.08)	1.44 (0.08)	1.47 (0.08)	1.52 (0.08)	1.54 (0.09)	0.70 (0.04)
Cs	11.33 (0.14)	11.07 (0.12)	10.23 (0.11)	10.08 (0.12)	10.63 (0.11)	11.54 (0.14)	0.99 (0.02)
Ba	595.5 (22.6)	576.0 (27.6)	552.5 (26.5)	547.4 (29.6)	577.0 (38.7)	598.9 (30.5)	145.6 (5.2)
La	47.75 (0.72)	45.53 (0.68)	44.33 (0.66)	42.39 (0.64)	44.08 (0.66)	46.55 (0.70)	3.90 (0.07)
Ce	93.5 (2.3)	90.7 (2.3)	83.9 (2.1)	81.4 (2.1)	90.9 (2.5)	95.7 (2.4)	6.6 (0.2)
Nd	43.7 (2.1)	46.8 (6.8)	36.7 (1.8)	36.6 (2.2)	39.8 (3.1)	42.4 (2.2)	4.0 (1.7)
Sm	9.2 (0.3)	8.3 (0.2)	8.3 (0.2)	8.2 (0.2)	8.8 (0.3)	8.8 (0.3)	0.8 (0.02)
Eu	1.81 (0.05)	1.76 (0.05)	1.64 (0.04)	1.62 (0.05)	1.70 (0.05)	1.84 (0.05)	0.17 (0.01)
Tb	1.30 (0.08)	1.28 (0.07)	1.19 (0.07)	1.18 (0.07)	1.14 (0.07)	1.33 (0.08)	0.11 (0.01)
Yb	4.12 (0.06)	4.00 (0.05)	3.64 (0.15)	3.60 (0.05)	4.03 (0.07)	4.28 (0.06)	0.51 (0.01)
Lu	0.57 (0.06)	0.51 (0.01)	0.43 (0.04)	0.50 (0.02)	0.55 (0.02)	0.57 (0.05)	0.07 (0.004)
Hf	4.04 (0.07)	4.12 (0.07)	3.74 (0.06)	3.71 (0.03)	3.79 (0.11)	4.35 (0.08)	1.10 (0.01)
Ta	1.34 (0.04)	1.30 (0.03)	1.23 (0.03)	1.21 (0.03)	1.33 (0.04)	1.40 (0.04)	0.26 (0.01)
Th	15.7 (0.3)	15.4 (0.3)	14.1 (0.3)	14.1 (0.3)	15.0 (0.3)	16.1 (0.4)	1.2 (0.03)
U	4.22 (0.14)	3.84 (0.13)	3.73 (0.13)	3.73 (0.12)	4.12 (0.17)	4.11 (0.12)	0.42 (0.03)

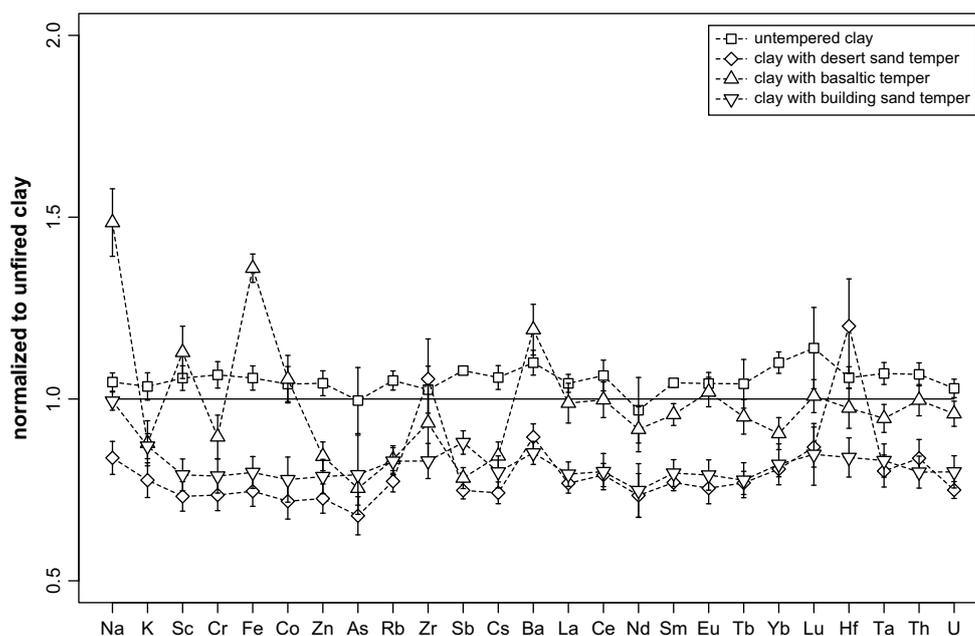


Fig. 1. The increase in all element concentrations in the untempered and fired tablets compared to the original, unfired clay.

contribute to the trace-element composition, as would be expected for quartz temper. Furthermore, by introducing the dilution factor f_0 , it is possible to mathematically remove the systematic spread introduced by dilution.

Additional to the multivariate filter, principal component analysis (PCA, see e.g. Jolliffe, 2002) was applied to the original as well as the dilution-corrected dataset. In PCA, the whole n -dimensional dataset, where each dimension represents an element concentration, is rotated in such a way as to maximize the variance along the first axis. After this first rotation, the next rotation maximizes the remaining variance along the second axis, perpendicular to the first. In this way, the variance of the dataset is distributed along all n axes in descending order of their variance explanation. After this procedure, a projection of the dataset onto the plane spanned by the first two axes, i.e. the most important principal components, should show possible structures in the dataset.

3. Results and discussion

3.1. Instrumental Neutron Activation Analysis

The numerical results of the INAA of the original clay (sample ClayOrig) as well as the fired tablets can be found in Table 1. Sample names starting with 'T' refer to the original clay, a leading 'S' stands for Sahara sand temper (see Table 2), 'B' denotes the basaltic temper (Table 3), and 'Bu' refers to the samples tempered with building sand (Table 4). The numbers 1, 2, and 3 denote the different firing temperatures, 900 °C, 1100 °C, and 1200 °C, respectively. Samples with names ending in 'D' have been taken using the fused alumina drill whereas a final 'G' in the sample name indicates a sample that has been broken off and ground. The uncertainties quoted are due to the statistical counting only.

Fig. 1 shows the mean elemental concentrations of all samples from the same paste normalized to the elemental concentration of the unfired original clay (sample ClayOrig, Table 1). The errorbars show one standard deviation, indicating that the difference introduced by the different burning temperatures and sampling

techniques are much smaller than the difference between the pastes. The relatively large error for Nd results from the large measurement error. Similarly, the larger error for Lu is a result of the measurement conditions. However, the large variation in the As concentrations is most probably due to the volatility of the element and its compounds, respectively. Considering that, from a petrological point of view, the As in the sample most probably occurs in the form of arsenopyrite (FeAsS) with a decomposition

Table 5

Average concentrations of elements M in $\mu\text{g g}^{-1}$ and spreads σ (root mean square deviation), also in percent of M, of group 1 (all clays and quartz-sand tempered samples) and group 2 (basaltic-sand tempered samples), individual samples corrected for dilution with respect to average grouping values, respectively.

	Group 1		Group 2		
	19 samples		6 samples		
	M +/- σ , %		M +/- σ , %		
Na	6912	± 696	10	10,604 ± 517	4.9
K	32,646	± 1645	5.0	31,900 ± 1643	5.1
Sc	18.7	± 0.73	3.9	24.5 ± 1.15	4.7
Cr	123	± 7.98	6.5	127 ± 8.26	6.5
Fe	23,810	± 766	3.2	37,256 ± 1104	3.0
Co	24.3	± 1.43	5.9	30.2 ± 1.99	6.6
Zn	173	± 7.15	4.1	170 ± 5.15	3.0
As	23.3	± 2.56	11	21.3 ± 0.96	4.5
Rb	190	± 6.53	3.4	178 ± 6.09	3.4
Zr	170	± 26.4	16	162 ± 14.6	9.0
Sb	1.53	± 0.097	6.4	1.33 ± 0.076	5.7
Cs	11.7	± 0.39	3.3	11.4 ± 0.15	1.3
Ba	638	± 30.6	4.8	801 ± 34.1	4.3
La	49.5	± 1.14	2.3	56.1 ± 2.18	3.9
Ce	98.9	± 2.50	2.5	111 ± 2.80	2.5
Nd	43.7	± 2.89	6.6	49.5 ± 2.93	5.9
Sm	9.41	± 0.30	3.1	10.3 ± 0.31	3.0
Eu	1.89	± 0.052	2.7	2.23 ± 0.060	2.7
Tb	1.37	± 0.080	5.8	1.51 ± 0.088	5.8
Yb	4.35	± 0.10	2.3	4.29 ± 0.076	1.8
Lu	0.58	± 0.026	4.6	0.63 ± 0.031	4.9
Hf	4.92	± 1.03	21	4.57 ± 0.11	2.5
Ta	1.41	± 0.039	2.8	1.48 ± 0.039	2.6
Th	17.0	± 0.93	5.4	18.8 ± 0.41	2.2
U	4.28	± 0.14	3.3	4.75 ± 0.17	3.6

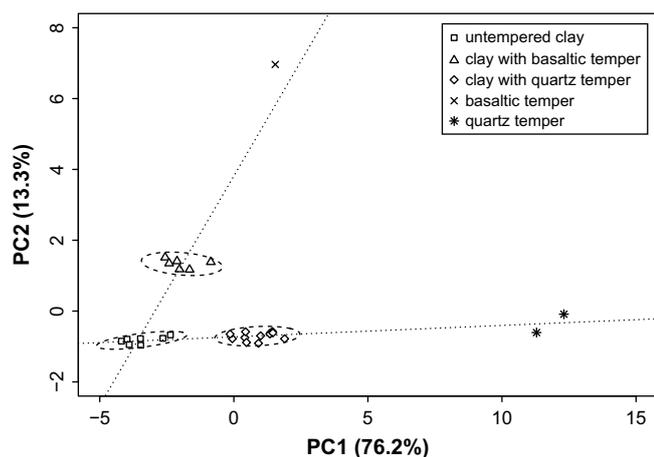


Fig. 2. Results of the Principal Component Analysis. As expected, the tempered samples lie on a straight line between the untempered clay and their respective tempers. The percentage of the total variance that is explained by the two principal components is 76.2% and 13.3%, respectively.

temperature of 570 °C, there should be no systematic difference between the samples fired at different temperatures, as even the lowest temperature of 900 °C is more than sufficient to mobilize the element, especially under oxidizing conditions. However, during the drilling process it is certainly possible to locally reach temperatures in excess of 500 °C, depending on the sharpness of the drill and the pressure applied. These considerations are supported by the data (see Tables 1–4), as the As values for the ground samples show less spread than the values for the drilled samples with the exception of the desert sand tempered samples, where the spread was constant. For the other elements, no significant influence by firing temperature could be observed, in accordance with literature (e.g. Cogswell et al., 1996; Kilikoglou et al., 1988; Riccardi et al., 1999).

Comparing the element concentration values of the unfired clay with the corresponding values of the fired clay tablets made from untempered paste, clearly shows that during firing water of crystallization and adsorbed water is removed from the sample whereas the drying process alone does not remove all the water contained in the clay. Thus all element concentrations in the fired samples are increased by a mean factor of approximately 1.05 (see Fig. 1).

The two pastes prepared with tempers that consist mainly of quartz, building sand and Sahara desert sand, show a very similar distribution in Fig. 1. The strong discrepancy in the Zr and Hf values indicates that the desert sand contained zircon ($Zr(Hf)SiO_4$) which was practically absent in the building sand. Due to the amount present, it was not surprising that zircon was not detected by XRD, but it could easily be found by optical microscopy in an additional investigation to check the size of the zircons. Since for all samples tempered with Sahara sand, the Zr and Hf values are consistently lower for the drilled samples than for the ground samples (ranging from 77% to 97%) it stands to reason that the drilling somehow removed the zircon crystals. During the drilling of the Sahara sand tempered samples it was observed that small crystals, or crystal fragments, were catapulted out of the sample. It can be assumed that the quartz grains that have a characteristically mottled surface due to aeolian transport are much better retained in the fired matrix than the zircon grains that exhibit a very smooth surface.

The samples tempered with basaltic sand show the largest deviations from the original untempered clay. This is the expected effect since the basaltic temper contains significant amounts of the elements measured.

3.2. Statistical analysis

To check the influence of the different tempers on the grouping of the samples, the complete dataset was grouped using the multivariate statistical filter developed by Beier and Mommsen

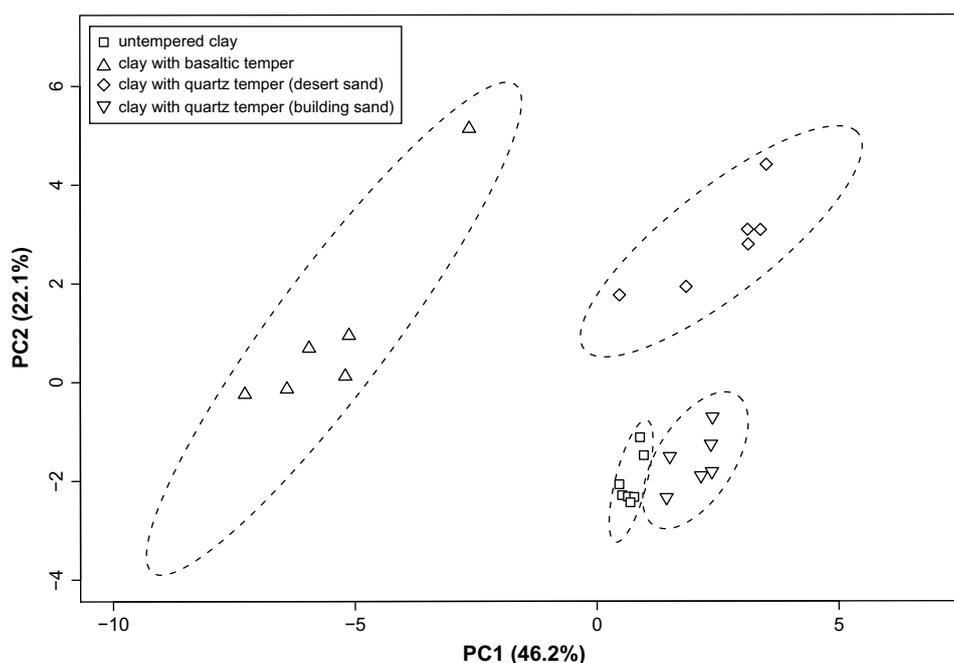


Fig. 3. Results of the Principal Component Analysis of the dilution-corrected dataset. By mathematically removing the dominant spread related to the dilution by various tempers, subtle differences like the two different quartz tempers become relevant. The percentage of the total variance that is explained by the two principal components is 46.2% and 22.1%, respectively, much less than in Fig. 2.

(1994). Dilution factors were calculated using all elements available. All samples were compared with each other resulting in two large groups. One group contained all samples tempered with basaltic sand (samples B1R, B2R, B3R, B1B, B2B, and B3B) whereas all other samples were grouped together. The samples of the first group showed dilution factors ranging from 0.96 up to 1.06 with respect to their mean.

The second group includes not only the samples made from the pastes with quartz temper but also the untempered clay samples, including the unfired clay. For the tempered samples, the dilution factors with respect to the group mean vary between 0.97 and 1.07, for the untempered samples the dilution factors vary between 0.73 and 0.79 averaging to 0.75.

In Table 5, the average concentration values of the two groups are shown. With minor exceptions, spreads are not much higher than the experimental uncertainties pointing to the fact, that it is the clay that determines provenance. The large spreads for Zr and Hf point to the possibility to form a subgroup of the desert sand tempered samples as already discussed. The larger spreads in Zr, Hf, and As have already been discussed. The larger spread in Na for the first group can be explained by the occurrence of sodium feldspar (albite) in the temper.

Fig. 2 shows the results of the principal component analysis of the unfiltered, original dataset. By representing samples as vectors with a dimension corresponding to the elements measured, a mixture between two samples corresponds to a linear combination of the two samples. Thus tempered samples lie on a straight line between the untempered clay samples and their respective tempers. This can also be expanded to the mixture of different pastes (see Schwedt and Mommsen, 2004). If the whole original dataset (excluding the pure temper samples) is considered to be systematically disturbed by dilution, it is possible to correct for this by performing a dilution correction for all samples with respect to the average of all samples, i.e. each sample is corrected for a hypothetical dilution with a single factor for all elements. After this correction, PCA can again be performed, as is demonstrated in Fig. 3. As can be seen by the relatively small percentage of the total variance that is explained by this PCA compared to Fig. 2, a large part of the spread introduced by dilution was removed. In this case the correction still clearly separates the samples with basaltic temper but moves the untempered and the quartz-tempered samples closer together. However, a separation between the two different quartz-dominated tempers (i.e. desert sand and building sand) is possible, showing the potential to distinguish even small subtle changes in the recipe that was used to create the paste.

4. Conclusion

The preparation of several different pastes from one source of levigated clay offers the possibility to investigate the influence of several tempers on the elemental concentration values of the respective paste under controlled conditions. The application of two different sampling methods and of three different firing temperatures furthermore made it possible to establish if the method of sampling and temperature of firing has significant impact on the final results.

The results of the INAA clearly show (Fig. 1) that the different firing temperatures or sampling methods have some influence on the results of the measurement, especially if volatile elements or compounds (As) are determined or hard crystals (Zr, Hf in the form of $Zr(Hf)SiO_4$) are lost during sampling. However, the influence exerted by the sampling methods and firing temperatures is significantly smaller than the difference introduced by different tempers, even if they are very similar in their composition, as the building and desert sand. Thus, since the firing temperature of

ancient pottery cannot be influenced, the sampling method applied has negligible influence on the feasibility for provenancing. However, the authors tend to prefer the method of breaking off small pieces of the sherd, although more laborious, not only because of the larger influence the drilling can have but also because a freshly cracked surface offers the possibility of microscopic investigations.

Looking at the results of the PCA (see Fig. 2), it can be clearly seen that the difference between various pastes, clay and tempers can be established by this method. However, a separation of the two quartz-dominated tempers is not possible with this traditional approach. However, if samples of possible tempers or other additions are available for analysis, the linear extrapolation between the temper and the tempered material could, in principle, lead to the establishment of the original clay source.

The application of the multivariate statistical filter to the grouping of the samples shows the great potential this method offers. Not only were all samples correctly associated, but due to the application of dilution factors, the quartz temper was ignored and the similarity (or 'provenance') to the untempered clay was clearly established. In the case of a highly influential temper like the basaltic sand, simple dilution correction cannot be sufficient to establish the source. However, it can be safely assumed that the use of a temper like basaltic sand (the only temper that led to a change in color of the finished product) was applied as a different recipe and therefore the separation from the other, quartz-tempered products is important. Furthermore by applying just a simple dilution correction, it is possible to distinguish the two very similar quartz-dominated tempers (see Fig. 3). This shows that by mathematically removing the spread introduced by dilution, even small differences in the recipes can be made visible with traditional multivariate methods.

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