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Pottery firing temperatures: a new method for determining the firing temperature of ceramics and burnt clay

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ABSTRACT

A new method for determining the maximum firing temperature of ceramics and burnt clay is presented. The technique relies on measuring the magnetic susceptibility on a step-wise re-fired sample. The validity of the method has been tested by determining firing temperatures of two sets of clay samples fired at temperatures ranging from 400 to 1000 °C. Aliquots of the same samples have been studied petrographically by optical microscopy on thin sections and analyzed by powder X-ray diffraction in order to monitor structural and mineralogical changes as a function of temperature. The method is demonstrated on samples from four geographically widely different sites and it is applied to a larger set of ceramics of Late (*ca.* AD 900–AD 1450) and Inca (*ca.* AD 1480–AD 1532) periods from the Northwestern Argentine region, dating to a limited period of time prior to the fall of the Inca Empire. The method is shown to be a powerful tool in revealing archaeological information about the change in firing technologies in the pre-Hispanic societies in the Andean area through time.

1. Introduction

There has been a long debate about how to determine the maximum firing temperature of archaeological ceramics and the archaeological meaning that can be ascribed to such data (see *e.g.* Gosselain, 1992; Livingstone Smith, 2001; Tite, 1995, 2008). It is agreed in the literature that the simplistic 'open firing' *versus* 'kiln firing' discussion leaves out much of the complexities of the firing procedure, although either of these assumptions may be viable or at least practical archaeological approximations. In general the analytical methods applied to determine firing temperatures involve establishing a relationship between the firing temperature and changes in mineralogy and possibly in the microstructure of the pottery, such as porosity, clay matrix, progressive sintering, and vitrification (*e.g.* Tite, 1995: 37–38). The maximum firing temperature experienced by a vessel is important archaeological information pertinent to understanding the technological procedures applied by the ancient potters (Rice, 1987; Rye, 1981). The maximum firing temperatures are relevant in relation to establishing which kiln technology was utilized in a society and in order to distinguish between such technologies either in space or in time. Examples of the archaeological uses of firing temperature information are the introduction of Spanish colonial reign in Peru (Chatfield, 2010), the continuity of clay source use in Italy (Terenzi et al., 2010), differences in geological clay sources in Bulgaria (Jordanova et al., 2001), and for assessing duration, firing temperature and oxygen tension in ceramics production in Italy (Benedetto et al., 2002).

The firing temperature of archaeological ceramics has been estimated by a number of different methods. Amongst the methods working on a larger temperature scale are: 1) observations of the thermal expansion and dilation of regularly shaped samples of the ceramics (Roberts, 1963; Tite, 1969); 2) measurements of the coercive force and saturation magnetization (Coey et al., 1976); and 3) X-ray diffraction measurements of the layer spacing in the clay mineral illite (Maggetti and Rossmanith, 1981). Other methods have also been invoked, such as the study of sintering and vitrification of the clay matrix by observing the microstructure by SEM (Chatfield, 2010; Maniatis and Tite, 1981; Musthafa et al., 2010; Velraj et al., 2008); monitoring the intensity of Mössbauer lines (Hayashida et al., 2003a, 2003b; Lumbreras

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et al., 2003; Shimada et al., 2003a, 2003b, 2003c, 2003d); the use of colour coordinates (Mirti, 1998; Mirti and Davit, 2004); ESR signal, penetration of high frequency radiation, and Vickers hardness. Comprehensive reviews are provided by Heimann and Franklin (1979) and Tite (1995, 2008). Disadvantages of several of the methods include limited accuracy and applicability over a limited temperature range only. In general, any method is also susceptible to the uncertainty introduced by the firing history. Besides the maximum temperature, this includes parameters such as the heating rate, duration of the firing, the cooling rate, and possibly also re-firing events. Furthermore, the temperature can be a complicated function of time, which may not necessarily constitute a good approximation to the preferred laboratory step-function.

This paper describes a novel method for the determination of the maximum firing temperature for ceramics and burnt clay. The method can be applied to samples as small as a few milligrams of archaeological ceramics. Results of the method have previously been reported (Rasmussen, 2003; Rasmussen and Hjermind, 2006), but the method itself has not been described and validated in detail. Here we report a validation experiment extending from 400 to 1000 °C from which we estimate an uncertainty of the method of ±25.8 °C (1 sigma). In order to further validate the method, it has been applied to 4 sherds from widely different archaeological sites around the world, and to 36 sherds coming from six different but closely spaced archaeological sites located in the Southern Abaucán Valley in the Province of Catamarca, Argentina.

2. Description of the method

The main raw material for ancient ceramic production is often clay, which consists of clay minerals mixed with a variety of other minerals. Upon firing they undergo characteristic reactions such as dehydroxylation, decomposition, and transformation. Precisely which stable and/or metastable neoformation phases are produced during the firing depends upon the mineralogy of the raw material, its grain-size distribution, maximum firing temperature, duration of the firing, and the redox conditions in the atmosphere of the kiln (Cultrone et al., 2004; Jordan et al., 2008; Moropoulou et al., 1995). The magnetic properties of a ceramic vessel often changes upon firing due to a combination of destruction of some of the original minerals and the occurrence and growth of neoformation mineral phases. A wide range of clay compositions are found worldwide, which makes the accurate determination of firing temperature inherently difficult. However, in most cases a change in the mineralogical assemblage is introduced upon firing and very often these changes cause a change in the magnetic susceptibility of the bulk sample. The magnetic susceptibility is a material parameter defined as the proportionality factor (χ) between the induced magnetization (J) and the externally applied magnetic field (H): $J = \chi H$. The magnetic susceptibility is dimensionless in the SI-unit system.

The size of the change in magnetic susceptibility is a function not only of which minerals are destroyed and which are formed, in each case depending on the passing of a specific threshold temperature, but also on the grain-size which the neoformation minerals grow to. Once the ceramic vessel is cooled from its maximum firing temperature, the processes of neoformation of minerals will normally not be reversed, and the high temperature mineral assemblage will to a large degree be preserved over archaeological time. It is therefore from the outset likely, if not completely predictable how, that the maximum firing temperature can be read off a curve of the magnetic susceptibility as a function of re-firing temperature.

2.1. Details of the method

A solid sample of ceramic material is dried for at least 24 h at 120 °C, cooled, weighed and measured in quadruple for magnetic susceptibility on a Kappabridge KLY-2 magnetic susceptibility meter. The susceptibility of the empty sample holder is measured and subtracted from the sample values. The Kappabridge is calibrated twice a day using a standard sample of known susceptibility. Generally the reproducibility is better than 1% relative and the limit of detection is ca. 1×10^{-6} SI-units. The average magnetic susceptibility is calculated using the sample weight and assuming a standard density of 2.5 g cm^{-3}. A constant density is assumed because it is inherently difficult to measure density of oddly shaped archaeological ceramic samples and because an error in the estimation of the density will only parallel-shift the curve of the magnetic susceptibility *versus* re-firing temperature by a fixed amount, which will not in any way affect the determination of the firing temperature as described below. The method has been used for samples between 10 mg and 5 g in weight. The measuring sequence starts by firing the sample in a Carbolite 1100 CWF oven at 200 °C (±1 °C) for 24 h, after which it is cooled to room temperature and measured in quadruple for magnetic susceptibility. This treatment is repeated in 20 °C steps from a temperature of 200 until ca. 1000 °C, for increasingly smaller times, ending up with a firing time of 30 min for temperatures above 900 °C.

The susceptibility data are subsequently plotted as a function of the re-firing temperature. Examples of such graphs are shown in Fig. 1; the samples are taken from widely different archaeological sites in Iran, Denmark, Argentina, and Israel. In the plot, the sudden discontinuity marks the point when the original maximum firing temperature is exceeded. A similar behaviour when superseding the original firing temperature, but with changes in the colour coordinates, has previously been reported by Mirti (1998). In order to establish this threshold temperature unambiguously and more accurately than just attempting to read it off the susceptibility *versus* temperature curve, the square of the first derivative is plotted as a function of stepwise re-firing temperature. The derivative is calculated as $(S_i - S_{i-1})/\delta T$, where S_i and S_{i-1} are two consecutive susceptibility measurements and δT is the temperature difference (in this study 20 °C). The squared derivative of the data is also shown in Fig. 1. The determined maximum firing temperatures are indicated with arrows in Fig. 1.

3. Results

3.1. Validating the method through an experimental approach

In order to validate the method, we have applied it to two sets of clay samples fired at specific temperatures in a laboratory oven, a Carbolite 1100 CWF with ±1 °C temperature control. For the two series (termed series 1 and A), the same yellow-burning 'blue' clay from the locality Solkær near Haderslev in Denmark was procured in the field and formed into 1 × 1 × 2 cm bricks, each pair (series 1 and A) incised with the firing temperature. The 14 samples were dried at 120 °C for 48 h, followed by another drying at 200 °C for 5 days in order to avoid cracking of the samples while firing to higher temperatures. The samples were then fired at temperatures 400, 500, 600, 700, 800, 900 and 1000 °C for progressively shorter times, reflecting the diminished need for equilibration at higher temperatures (see Table 1). The fired samples were then divided into 4 subsamples, each being weighed with 0.1 mg accuracy, and on average weighing 1.4 g.

The samples from Series 1 were measured for firing temperature with stepwise re-firing times of ca. 16 h per step from 200 to 600 °C. From 600 to 700 °C the re-firing time was 4 h, from 700 to 850 °C it

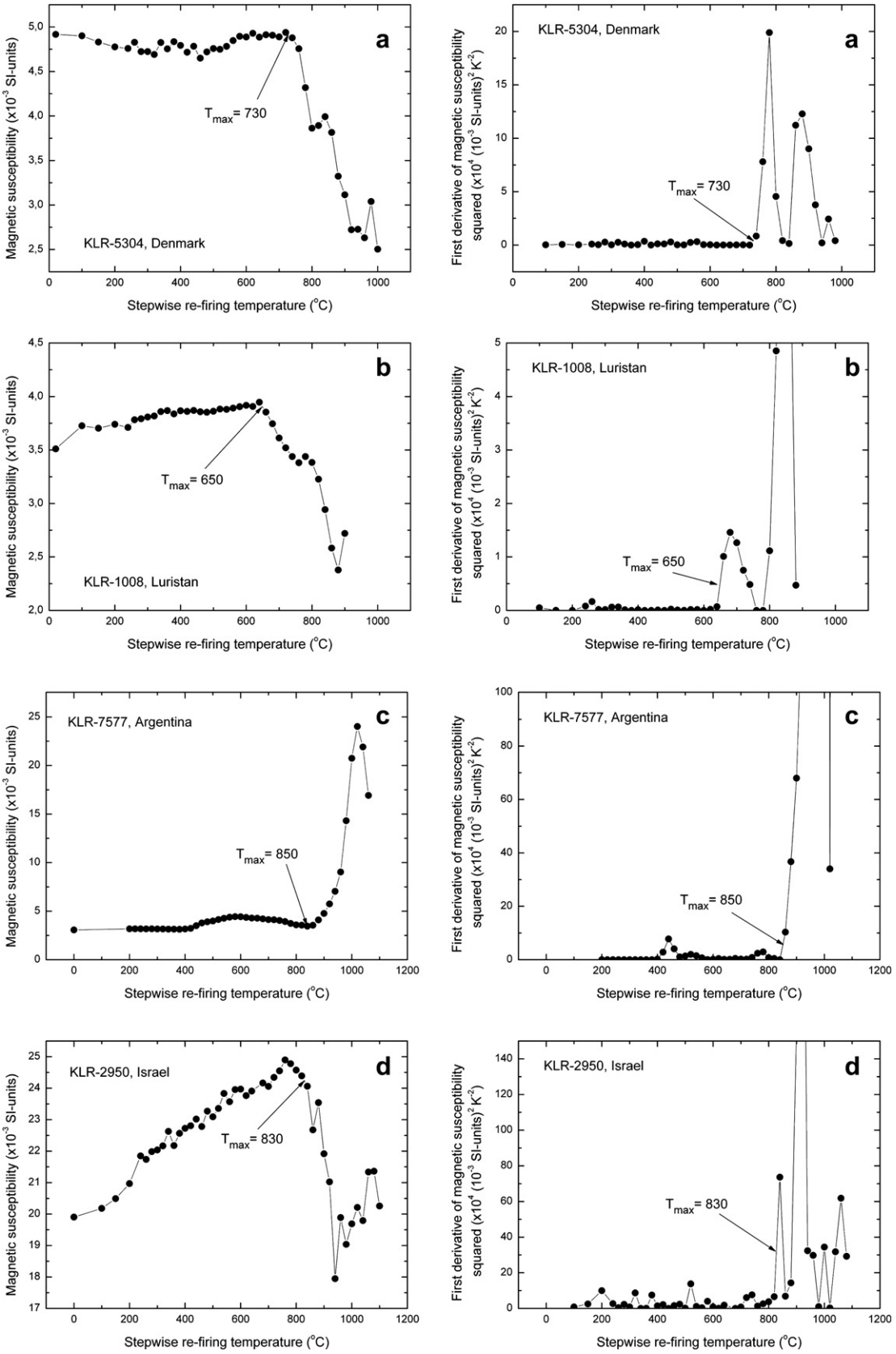

Fig. 1. Examples of experimental determination of the maximum firing temperatures. Four samples have been selected from archaeological sites from widely different geological and geographical settings: (a) a sherd from a Medieval vessel from Viborg, Denmark; (b) an iron age sherd excavated by Henrik Thrane in the 1950's in Luristan, present day Western Iran; (c) a sample of Inca ceramics from the Catamarca region in Northern Argentina; (d) sample of a scroll jar found in locus 41 in Qumran, on the West Bank of the Dead Sea. Left diagrams show the measured magnetic susceptibility as a function of stepwise re-firing temperature. Right diagrams show the squared first derivative calculated from the data in the left diagram. The maximum firing temperatures are determined as the first large deviation from zero of the squared first derivative and are indicated with arrows.

Table 1
Results of the validation test for the fired clay samples. The data correspond to the plots shown in Fig. 3. The uncertainty of the method is determined as a square sum deviation of the fired and the experimentally determined firing temperatures and turns out to be ±25.8 °C.

Sample no.	Firing temperature °C	Firing time (h)	Measured firing temperature °C
KLR-7937	400	24	390
KLR-7938	400	24	390
KLR-7939	500	23	490
KLR-7940	500	23	490
KLR-7941	600	6	570
KLR-7942	600	6	570
KLR-7943	700	6	730
KLR-7944	700	6	730
KLR-7945	800	2	830
KLR-7946	800	2	830
KLR-7947	900	2	890
KLR-7948	900	2	890
KLR-7949	1000	2	990
KLR-7950	1000	2	990

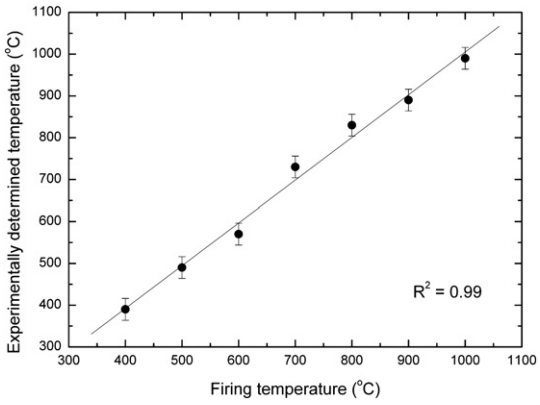

Fig. 3. Plot of pre-firing temperatures against the experimentally determined firing temperatures listed in Table 1. Each plotted point consists of two measuring points (identical measurements in all cases). The straight line is a linear regression line, and the correlation coefficient is $R^2 = 0.99$. The 1 standard deviation uncertainty is ±25.8 °C, which is shown as error bars.

was 2 h, from 850 to 950 °C 1 h, and from 950 to 1060 °C the stepwise re-firing time was 0.5 h. After the completion of the first series a complete replication of this procedure was carried out with the samples of Series A. The determined firing temperatures are listed in the last column of Table 1, and an example of the data is shown in Fig. 2, where both the magnetic susceptibility and the squared first derivative of the magnetic susceptibility are plotted against the stepwise re-firing temperature. The original firing temperature is easily identifiable in both plots (indicated by arrows). Fig. 3 shows the firing temperatures determined in this way as a function of the known firing temperatures. The straight line is a regression line calculated using the data from both series of samples simultaneously. The correlation coefficient of $R^2 = 0.99$ and the average uncertainty (1 sigma) of ±25.8 °C are quite satisfactory, especially when compared to the uncertainties of the previously used methods for determining firing temperatures of ceramics.

In order to examine the mineralogical changes in the samples of the artificial re-firing series, aliquots were subjected to powder X-ray diffraction (PXRD) analysis. The data were collected on a PANalytical XPert PRO diffractometer using Ni-filtered CuKα-radiation (tube voltage 45 kV, tube current 40 mA, average wavelength 1.5418 Å) and a solid-state PIXcel detector. The sample was placed in a stainless steel holder, and data were collected in Bragg-Brentano (reflection) geometry, with the sample rotated at 2 revolutions per second to minimise preferred orientation. Data were collected over the range 5–70 degrees 2-theta using a continuous scan for a total data collection time of 10 min. A selection of the PXRD patterns is shown in Fig. 4 and the main mineral phases are listed in Table 2.

Thin sections produced from another set of aliquots were studied petrographically. The minerals identified by optical microscopy are listed in Table 2 and optical micrographs of six of the test samples are shown in Fig. 5. In a sample only tempered at a temperature of 200 °C (KLR-7933) muscovite, microcline, Na-feldspar, and calcite were identified by optical petrography. These phases have disappeared in the samples fired at higher temperatures, i.e. 900 °C (KLR-7947 and 7948) and 1000 °C (KLR-7949 and 7950), where we identify ghelenite, wollastonite, akermanite, and diopsides, all of which are neoformation mineral phases (Molera, 1991, 1996; Bearat, 1992; Buxeda i Garrigós and Cau Ontiveros, 1995; Cultrone et al. 2001). The PXRD patterns support and complement the petrographic observations, showing clearly the destruction of calcite at higher firing temperatures and the accompanying rise of gehlenite and wollastonite (Fig. 6).

The nucleation and growth of hematite has previously been reported in pottery production using a mixture of clay and calcite (Duminuco et al., 1998; Dondi et al., 1999; Nodari et al., 2007),

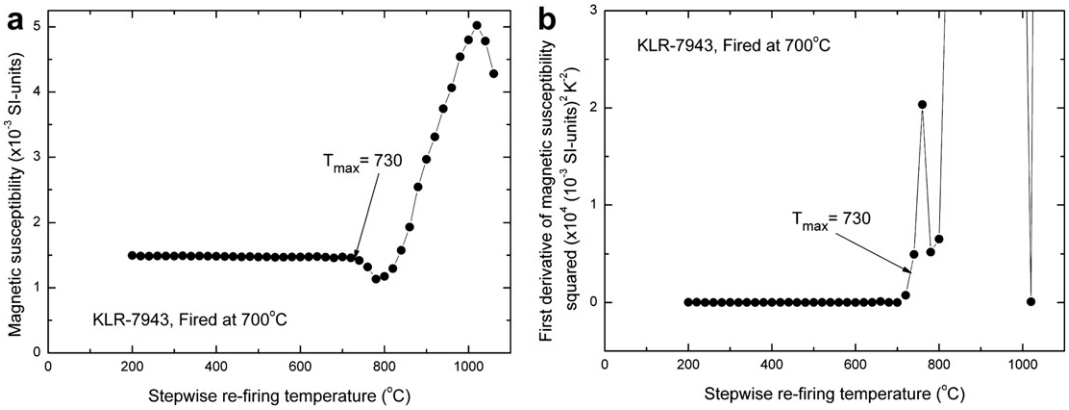

Fig. 2. a and b. Example of the determination of the maximum firing temperature of a validation test sample fired at 700 °C in the laboratory as part of the verification of the method. Left diagram shows the measured magnetic susceptibility as a function of stepwise re-firing temperature. Right diagram shows the squared first derivative calculated from the data in the left diagram. The maximum firing temperature is determined as the first large deviation from zero of the squared first derivative and is indicated with arrows.

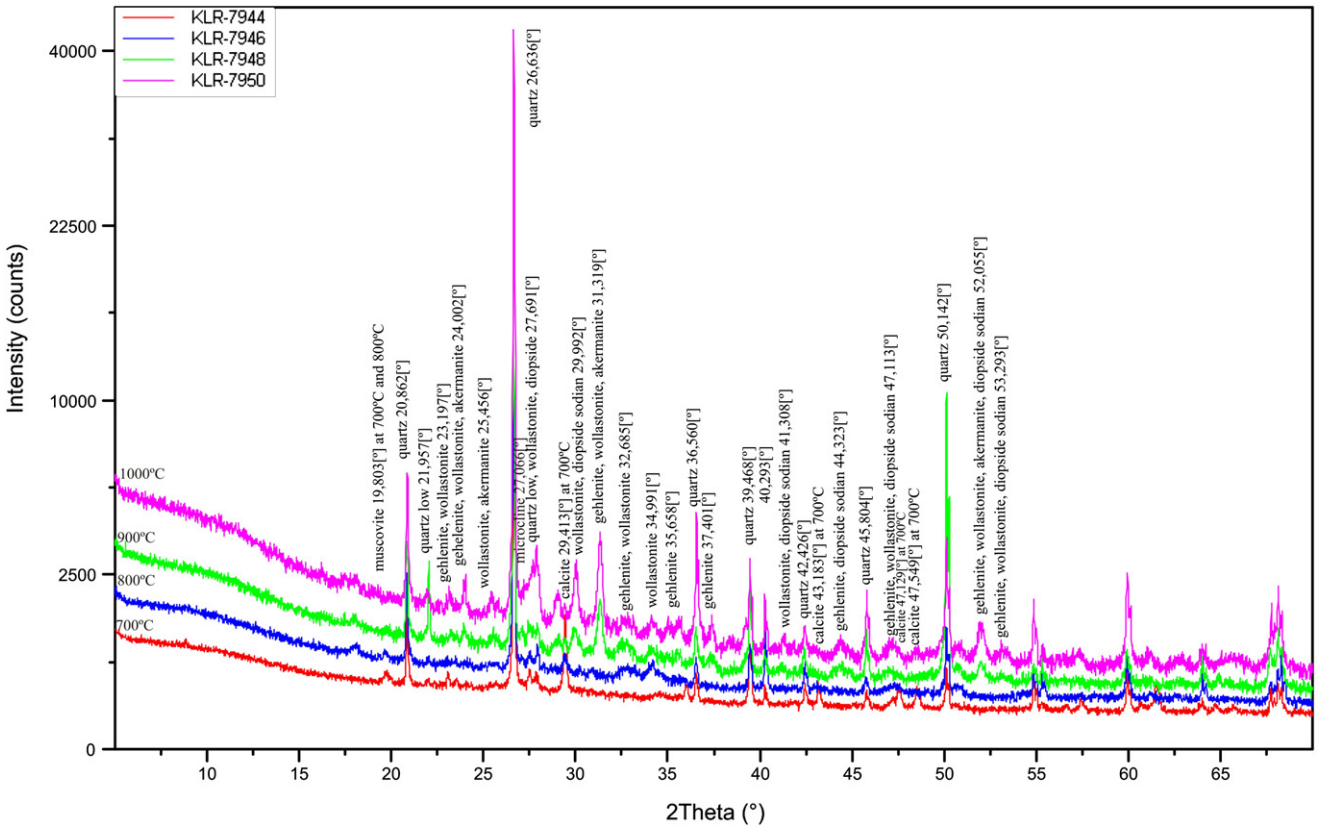

Fig. 4. Powder X-ray diffraction patterns of four test samples fired at 700 (KLR-7944), 800 (KLR-7946), 900 (KLR-7948), and 1000 °C (KLR-7950). Gehlenite, wollastonite, akermanite and diopsides are present as neoformation phases at the highest temperatures (900° and 1000 °C), whereas it is observed that calcite and muscovite have disappeared.

a composition that is very similar to the raw material used in this experiment. In the case of our test series, however, it is evident from the PXRD patterns that hematite (and also magnetite) is not observed, at least to the detection limit of the technique, which is estimated conservatively to be 2–3%. When both calcite and Fe are present during firing, it is also established that the neoformation minerals gehlenite ($Ca_2Al(AlSi)O_7$), wollastonite ($CaSiO_3$), and diopside ($CaMgSi_2O_6$) can incorporate Fe. In wollastonite this does not alter the magnetic susceptibility, but in gehlenite the incorporation of Fe can increase the magnetic susceptibility drastically. As shown by Nöller and Knoll (1983), the magnetic susceptibility of Fe-gehlenite can reach 36.5×10^{-3} (in emu), which is in agreement with the measured susceptibility for the test samples in this study. The mineralogical origin for the change in magnetic susceptibility in the case of our test series is therefore thought to be the gradual formation of Fe-gehlenite, possibly augmented by small amounts of neoformation hematite. Both minerals occur only in relatively minor amounts – gehlenite in amounts sufficient to be detected by PXRD, and hematite, if it is present at all, below the detection limits of that technique.

3.2. Archaeological application of the method

Concerning the existence of any mineralogical or other constraints on the applicability of the method, we note that we have tested it so far on samples from over fifty different archaeological sites and only in a few percent of the cases has the method failed to show any sudden change in magnetic susceptibility. Fig. 1 shows examples of maximum firing temperatures determined on samples originating from widely different geographical areas and geological conditions: Denmark, Israel, Luristan (Iran), and Argentina. In each case a maximum firing temperature can easily be found (indicated with arrows).

To examine further the applicability of the method, we have determined firing temperatures for a series of ceramic samples ($N = 36$) from the Northwestern Argentine region, where we for archaeological reasons expect to find two different kiln technologies. The Late Period in Northwestern Argentina is traditionally characterized as a time of marked regional development, increased socio-political complexity, inequality, economic stratification, and an increasing number of internal conflicts (warfare) (González, 1977; González and Pérez, 1972; Ottonello and Lorandi, 1987; Raffino, 1983, 1991; Tarragó, 2000). Craft specialization is widely represented in the archaeological record of the pottery production (De La Fuente, 2011a, 2011b). Late Period ceramic forms in the Southern Abaucán Valley, Catamarca, are mainly bowls, *ollas*, and funerary urns. These ceramics are well-fired in fully oxidizing atmospheres mostly through the utilization of kilns (De La Fuente, 2011a; Feely et al., 2010). The arriving of Incas in the region led to a reorganization of the pottery production visible through new forms of labor (*mit'a*), the introduction of extremely standardized new forms such as *aríbalos* (jars) and plates, together with the application of different ceramic paste recipes, and probably the use of firing procedures different than those used before by the ancient potters of the Late Period (Espinoza Soriano, 1970, 1975, 1987; D'Altroy, 1992; D'Altroy and Williams, 1998; Hayashida, 1999; Murra, 1980, 1982; Spurling, 1992). Like in the Late Period, Inca pottery is generally well-fired in fully oxidizing atmospheres and kilns appear to have been located near the Inca residential sites (De La Fuente, 2011a).

The samples used in this study are from six different but closely spaced Late (*ca.* AD 900–AD 1450) and Inca (*ca.* AD 1480–AD 1532)

Table 2
Main mineral inclusions and rock fragments identified by optical microscopy in thin sections and mineral phases by XRD of a selection of fired test samples.

Lab no.	Temp. °C.	Minerals identified in thin-sections	Mineral phases identified by PXRD
KLR-7940	500	*Mineral and rock fragment inclusions* quartz: mostly fractured, size fine/medium, very abundant; plagioclase: albite, size fine, trace; calcite (primary): very coarse, trace; argillaceous inclusions: size coarse/very coarse, presence; igneous rock fragments: granites, size medium, trace; amphibole: hornblende, size very fine, trace; *Fabric* Matrix: anisotropic, formed by clay aggregations (2.5 YR 3/8); % inclusions: 10%; Distribution: fair; Pores: elongated	quartz, muscovite, microcline (K feldspar), calcite, feldspar (Na-component), illite
KLR-7941	600	*Mineral and rock fragment inclusions* quartz: size medium/coarse, very abundant, rounded; plagioclase: size fine/medium, moderated, some specimens altered to sericite; argillaceous inclusions: size fine, presence; microcline (K feldspar): size fine, presence; igneous rock fragments: size fine/medium, presence; pyroxene: size fine, presence; calcite (primary): size very coarse; trace; opaque minerals: size fine, trace; *Fabric* Matrix: anisotropic, formed by clay aggregates and very fine quartz (2.5 YR 3/7); % inclusions: 15%; Distribution: poor; Pores: elongated	quartz, muscovite, calcite, microcline (K feldspar), feldspar (Na-component), traces of illite
KLR-7944	700	*Mineral and rock fragment inclusions* quartz: size medium/coarse, very abundant, very rounded; plagioclase: size fine, moderated with inclusions altered to sericite; muscovite: size very fine, trace; igneous rock fragments: size medium/coarse, presence; amphibole: brown hornblende, size fine/medium, trace; calcite(primary): size coarse/very coarse, trace; pyroxene: size very fine, trace; *Fabric* Matrix: partially isotropic, formed by clay aggregates (5 YR 4/8); % inclusions: 20%; Distribution: poor; Pores: elongated and sub-rectangular	quartz, muscovite, calcite, microcline (K feldspar), feldspar (Na-component), diopside sodian
KLR-7946	800	*Mineral and rock fragment inclusions* quartz: size fine/medium, mostly rounded, very abundant; plagioclase: size very fine/fine, presence; microcline (K feldspar): size medium, trace; igneous rock fragments: size fine/medium, trace; amphibole: brown hornblende, size fine/medium, trace; calcite: size medium/coarse, trace; argillaceous inclusions: size fine, trace; *Fabric* Matrix: anisotropic, partially micaceous (2.5 YR 4/10); % inclusions: 10%; Distribution: fair; Pores: elongated with microfractures.	quartz, muscovite, calcite, microcline (K feldspar), anorthite, feldspar (Na-component), diopside
KLR-7948	900	*Mineral and rock fragment inclusions* quartz: size fine/medium, very abundant, fractured; igneous rock fragments: granites, size medium, trace; argillaceous inclusions: probably sandstone, size medium, trace; plagioclase: albite, size fine/medium, some specimens altered to sericite; amphibole: brown hornblende, size fine, trace; *Fabric* Matrix: isotropic, with very fine quartz at the bottom (5 YR 6/12); % inclusions: 15%; Distribution: fair; Pores: elongated.	quartz, ghelenite, wollastonite, akermanite, anorthite, diopside, quartz low, traces of hematite
KLR-7949	1000	*Mineral and rock fragment inclusions* quartz: size medium/coarse, very abundant, mostly rounded −there are polycrystalline quartz inclusions; plagioclase: size very fine, presence; igneous rock fragments: size medium/coarse, trace; amphibole: size fine, presence; microcline (K feldspar): size fine, trace; *Fabric* Matrix: isotropic, formed by clay aggregates (5 YR 6/10); % inclusions: 10%; Distribution: poor; Pores: elongated.	quartz, ghelenite, wollastonite, akermanite, diopside, quartz low, traces of hematite
KLR-7950	1000	*Mineral and rock fragment inclusions* quartz: size fine/medium, very abundant, low sphericity, and very fractured; plagioclase: size fine, moderate; microcline (K feldspar): size fine/medium, presence; amphibole: hornblende, size fine, presence; pyroxene: size fine, trace; argillaceous inclusions: size medium, trace; *Fabric* Matrix: isotropic (5 YR 6/10); % inclusions: 15%; Distribution: fair; Pores: elongated.	quartz, ghelenite, wollastonite, akermanite, diopside, quartz low, traces of hematite

References: Size (Orton et al., 1993; Wentworth scale (φ scale) modified from Folk, 1965); % abundance (semi-quantitative): very abundant (40–60%), abundant (20–30%), moderate (10–20%), presence (5–10%), and trace (<1%); % inclusions (Mathew et al., 1991); Distribution (Barraclough, 1992); Matrix description (Freestone, 1991).

period archaeological sites geographically located in the southern sector of Abaucán valley in the Province of Catamarca (see KMZ file-link for positions). Costa de Reyes N° 5 is an Inca site, known as a *tambo*, connecting with several other sites through the Inca road, while SaCat12, SaCat13, and CV2 are Late Period sites with abundant surface finds of ceramics. At SaCat13 Inca ceramics are also present as surface finds (De La Fuente, 2008; De La Fuente et al., 2010). Río Colorado is an Early Period locality with more than 10 archaeological sites. Inca ceramic forms found are *aríbalos* (flared-rim jars) and plates, while Late Period forms are mainly bowls and funerary urns (Fig. 7). Most of the Inca sherds belong to a specific typological category named Inca Provincial, but two Inca sherds,

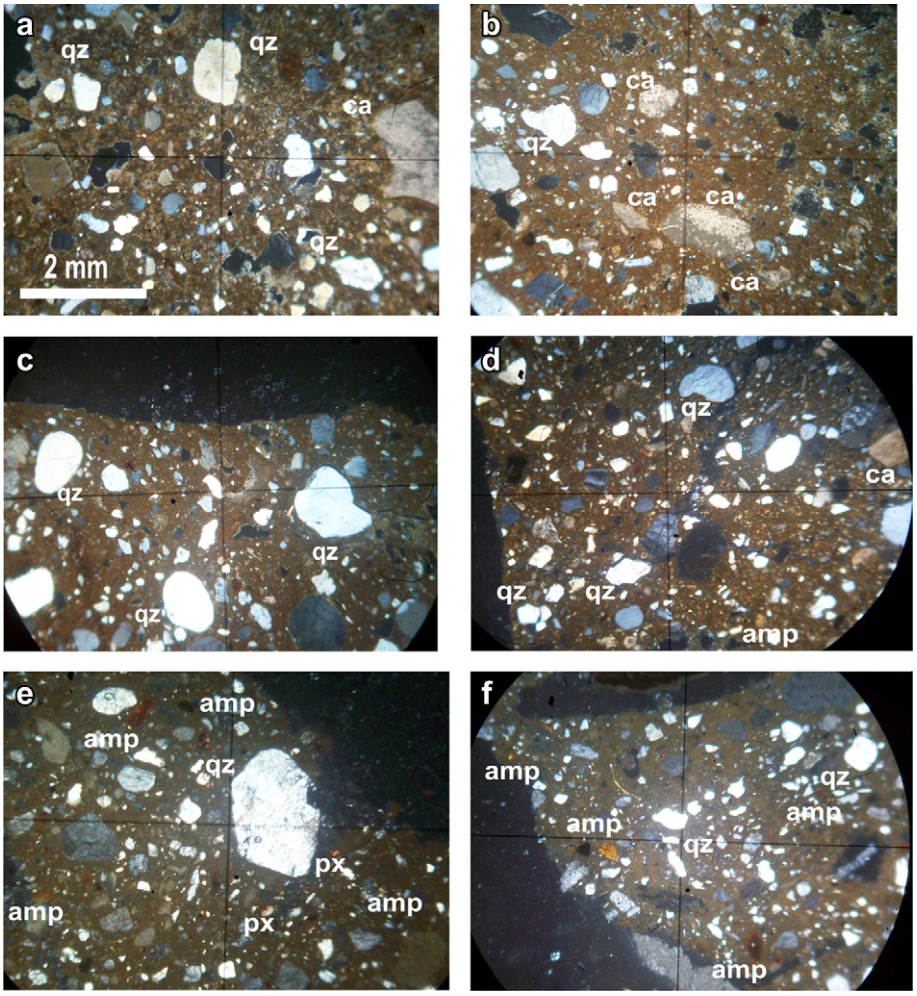

Fig. 5. Optical micrographs of thin-sections of six of the test samples; (a) KLR-7941 (600 °C); (b) KLR-7944 (700 °C); (c) KLR-7944; (d) KLR-7946 (800 °C); (e) KLR-7948 (900 °C); (f) KLR-7950 (1000 °C). Minerals identified by optical microscopy and main characteristics of the fabrics are listed in Table 2.

designated Inca-Diaguita Chileno and recovered at Costa de Reyes N° 5, are non-local ceramics coming probably from Chile.

An extensive ceramic petrology study carried out on 150 Inca and Late Period sherds showed mainly the presence of felsic minerals (quartz, alkali, and plagioclase feldspars), and igneous rock fragments (granite), complemented with muscovite and biotite minerals, few volcanic rock fragments (mainly andesite), accessory minerals like amphibole and clinopyroxene, and opaque minerals such as hematite and magnetite (De La Fuente, 1999, 2004, 2011a; see also Chatfield, 2010; and Hayashida et al., 2003b for descriptions of Inca ceramics from Peru). Additionally, for Inca ceramics we determined the presence of calcite (primary and secondary calcite) and the unique presence of grog (De La Fuente, 2011a). Preliminary multi-elemental chemical characterization by NAA shows that the ceramics are non-calcareous ceramics, with Inca ceramics forming a chemically very homogeneous group, and with the presence of some compositional variation for the Late Period sherds (De La Fuente et al., in press).

The maximum firing temperatures determined are listed in Table 3. For one sample only (DLF-038) the data were inconclusive and no firing temperature could be determined, which is probably due to a complex thermal history or one or more re-firing events. As pointed out also by Heimann and Franklin (1979), Livingstone Smith (2001), and Tite (2008), the precise functionality of the heating and cooling history, and any additional firing events to comparable or even higher temperature than the initial firing temperature, must influence the results of any method for determining firing temperature. As can be seen in Fig. 8, the distribution of firing temperatures shows two distinct peaks: one at 830 °C and one at 950 °C. Archaeological reasons for the presence of two distinctly different firing temperatures could be many, *e.g.* burning in two different types of kilns (Shimada et al., 1994, 2003b; Wagner et al., 1999; Hayashida et al., 2003b), the use of different types of fuel such as wood, grass, straw or dung (Sillar, 2000a, b), different people or families operating the same kiln, *etc.* In general terms it is likely that the firing temperature experienced by a vessel is dependant on the position it had in the kiln during firing, estimated by Mirti (1998) to be as large as 50 or even 100 °C (cf. Gosselain, 1992; Livingstone Smith, 2001). However, it seems unlikely that two distinct peaks 120 °C apart can result from inhomogeneous kiln temperatures. We therefore believe that the two peaks arise from two different firing practices.

4. Discussion

It could be asked if this method can be applied to clays with compositions other than those resembling the South Danish clay of the test series, the examples from the Middle East, and the Andean ceramics tested here. The prerequisite for the method to work is that one or more minerals with a sizable magnetic susceptibility are

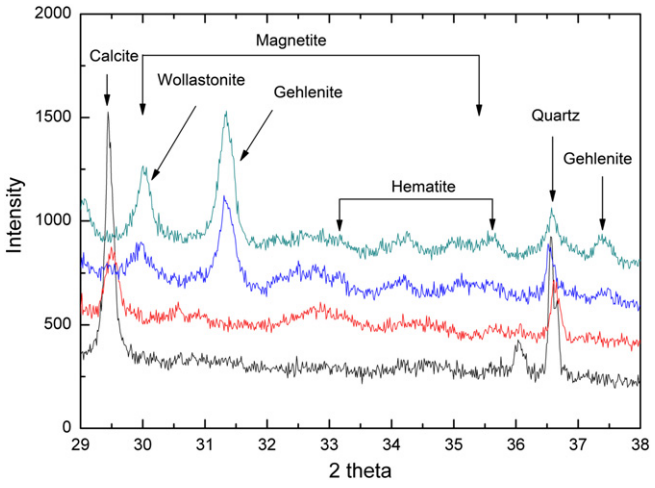

Fig. 6. Powder X-ray diffraction patterns in the range 29–38 degrees 2-theta of the test series samples fired at 700, 800, 900, and 1000 °C. From the bottom up: KLR-7943 (700 °C, black curve), KLR-7945 (800 °C, red), KLR-7948 (900 °C, blue), and KLR-7949 (1000 °C, green). The positions expected for peaks of calcite, wollastonite, magnetite, gehlenite, hematite and quartz are indicated with arrows. Gehlenite and wollastonite are neoforming minerals whereas calcite is disappearing with increasing temperature. There is no indication of magnetite in any of the patterns. Peaks attributable to hematite are hardly discernable, and it can be present only in minute amounts at elevated temperatures, if at all.

either destroyed or neo-formed upon firing. The destruction of calcite or the neoformation of wollastonite, for instance, will not lead to a significant change in magnetic susceptibility. However, if hematite or Fe-gehlenite (as seen in South Danish example) or any other Fe-rich mineral exhibiting a magnetic susceptibility is formed, the method will be applicable. Similarly, the method will work if for instance Fe-rich carbonates or oxides, like *e.g.* siderite or goethite, are destroyed upon firing. Even in cases without destruction or neoformation of minerals, processes such as re-crystallization, crystal size growth or magnetic domain growth are likely to provide enough change in magnetic susceptibility for the method to work (see Shimada et al., 2003a, 2003b). There can undoubtedly be found examples, where the method will fail: *e.g.* for samples consisting of pure calcite, pure quartz, or haphazard mineral assemblages where the decrease in susceptibility from the destruction or neoformation of a mineral is counterbalanced by an exact similar increase in magnetic susceptibility of other minerals. However, such cases must be rare. We have tested the method on ca. 50 different sites in Europe, the Middle East and in South America and encountered only a few percent of the samples where the method has failed to yield a sudden change in susceptibility. Consequently, we believe that the method is applicable for the majority of archaeological fired clays.

What, then, is the archaeological usefulness of establishing the firing temperature? First, it must be noted that under normal conditions the original firing temperature in a kiln is higher than the temperature exposure over a cooking fire or in a cooking oven (Chatfield, 2010; Hayashida et al., 2003b; Livingstone Smith, 2001; Rice, 1987; Rye, 1981; Tite, 1995, 1999, 2008). Therefore the archaeological conjectures revolve around the technology and conditions in the kiln producing the ceramics. In the ideal case a method like the present can help discriminating ceramics from different kilns either at different sites or at different times, and thus act as an indirect tool for provenancing ceramics. On a larger time scale the method can be used to track developments of kiln technologies.

Andean pottery was mostly fired using open methods such as open firings or bonfires (Chatfield, 2010), although some cases have been reported on the utilization of kilns, simple kilns, and insulated bonfires (Feely et al., 2010; Shimada et al., 1994, 2003b; Wagner et al., 1999; Hayashida et al., 2003b). The advantages and disadvantages of these opposed firing procedures have been stated and

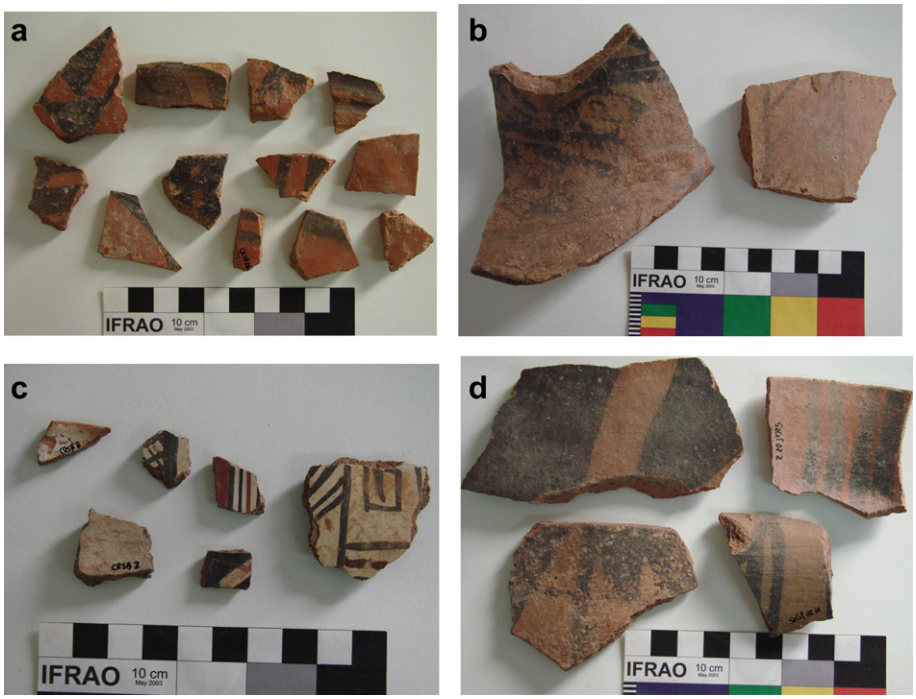

Fig. 7. Late and Inca periods archaeological ceramics analysed in this study: (a) and (b) Inca Provincial sherds, Costa de Reyes N° 5, Sector B; (c) Inca-Diaguita sherds, Costa de Reyes N° 5, Sector B; (d) Late Period sherds, Sanagasta Culture, SaCat12 and SaCat13 sites.

Table 3
Results of the determination of maximum firing temperatures of the samples from the Catamarca region in Northern Argentina. DLF-No and KLR-No are field and laboratory designations. The uncertainty of the measured firing temperatures are ca. ±25 °C (1 sigma). The fourth column indicates the archaeological site, and the last column describes the type of ceramics.

KLR-no.	DLF-no.	Measured firing temperature [°C]	Site	Sample description
INCA 1 series				
KLR-7573	DLF-005	830	SaCat 12	Sanagasta, rim frag., bowl
KLR-7574	DLF-006	950	SaCat 12	Sanagasta, rim frag., bowl
KLR-7575	DLF-015	830	SaCat 12	Sanagasta, rim frag., bowl
KLR-7576	DLF-011	830	SaCat 12	Sanagasta, rim frag., urn
KLR-7577	DLF-032	870	Rio Colorado	Saujil, rim frag., olla
KLR-7579	DLF-038	undetermined	Rio Colorado	Saujil, rim frag., bowl
KLR-7580	DLF-039	950	Rio Colorado	Saujil, rim frag., bowl
KLR-7600	DLF-104	950	Costa de Reyes n 5 (CRSB)	Sanagasta, body frag., urn
KLR-7601	DLF-105	890	Costa de Reyes n 5 (CRSB)	Inca, body frag., aríbalo
KLR-7602	DLF-108	870	Costa de Reyes n 5 (CRSB)	Inca, body frag., aríbalo
KLR-7603	DLF-109	950	Costa de Reyes n 5 (CRSB)	Inca, body frag., aríbalo
KLR-7604	DLF-110	850	Costa de Reyes n 5 (CRSB)	Inca/Belén, body frag., aríbalo
KLR-7605	DLF-116	910	Costa de Reyes n 5 (CRSB)	Inca, body frag., aríbalo
KLR-7606	DLF-123	790	Costa de Reyes n 5 (CRSB)	Inca/Diaguita Chileno, body frag., aríbalo
KLR-7607	DLF-127	870	Costa de Reyes n 5 (CRSB)	Inca, body frag., aríbalo
KLR-7608	DLF-131	770	Costa de Reyes n 5 (CRSB)	Inca, body frag., aríbalo
KLR-7609	DLF-132	890	Costa de Reyes n 5 (CRSB)	Belén, body frag., urn
KLR-7610	DLF-134	830	Costa de Reyes n 5 (CRSB)	Sanagasta, body frag., bowl
INCA 2 series				
KLR-7584	DLF-056	950	SaCat 13	Inca, body frag., aríbalo
KLR-7585	DLF-057	950	SaCat 13	Inca, body frag., aríbalo
KLR-7586	DLF-059	930	SaCat 13	Inca, body frag., aríbalo
KLR-7587	DLF-063	950	SaCat 13	Inca, body frag., plate
KLR-7611	DLF-139	810	Costa de Reyes n 5 (CRSB)	Sanagasta, body frag., urn
KLR-7612	DLF-140	830	Costa de Reyes n 5 (CRSB)	Sanagasta, body frag., bowl
KLR-7613	DLF-141	830	Costa de Reyes n 5 (CRSB)	Sanagasta, body frag., urn
KLR-7614	DLF-143	950	Costa de Reyes n 5 (CRSB)	Sanagasta, rim frag., urn
KLR-7615	DLF-148	1030	Costa de Reyes n 5 (CRSB)	Sanagasta, rim frag., bowl
KLR-7621	DLF-175	850	Costa de Reyes n 5 (CRSA)	Inca/Diaguita Chileno, body frag., aríbalo
KLR-7632	DLF-204	890	CV2	Sanagasta, body frag., bowl
KLR-7633	DLF-211	930	CV2	Sanagasta, body frag., urn
KLR-7634	DLF-212	950	CV2	Sanagasta, frag. base, urn
KLR-7635	DLF-216	870	CV2	Sanagasta, rim frag., bowl
KLR-7636	DLF-220	830	CV2	Sanagasta, body frag., bowl
KLR-7637	DLF-226	950	CV2	Sanagasta, body frag., bowl
KLR-7638	DLF-227	930	CV2	Sanagasta, body frag., bowl
KLR-7639	DLF-228	830	CV2	Belén, body frag., bowl

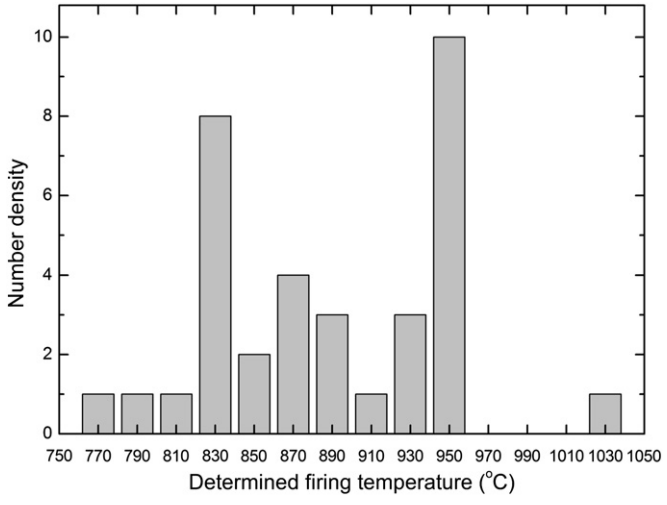

Fig. 8. Experimentally determined maximum firing temperatures of 35 ceramic samples from six different sites in Northern Argentina (data in Table 3). The uncertainty in the determination is ±ca. 25 °C. The distribution shows two distinct peaks, at 830 and 950 °C.

discussed according to the behaviour of a series of variables: (1) maximum temperature achieved, (2) the proximity of fuel to pots, (3) the cooling process, (4) firing atmosphere, (5) time at the maximum temperature during the firing, (6) and the optimum firing temperature or the maturation temperature (Chatfield, 2010: 727–730; Tite, 1995, 1999, 2008). Variables such as type of fuel, distribution of the pots during the firing, different types of firing structures, firing atmospheres have also been studied and discussed in light of ethnoarchaeological and ethnographic examples in the Andes (see Sillar, 2000a, b; cf. Gosselain, 1992; and for detailed cases in Africa: Livingstone Smith, 2001).

In Northwestern Argentina much of the larger ceramics are reported as fired in open firings or bonfires, although in some geographical areas the use of kilns, simple kilns or insulated bonfires appears to be more common than previously thought. Pottery kilns of different shapes have been reported throughout the Abaucán Valley (Feely et al., 2010, in press; De La Fuente, 2011a). The results of maximum firing temperatures obtained in this study show two distinct maximum temperature peaks at 830 °C and 950 °C. From Fig. 8 and Table 4 it can be seen that maximum firing temperature divides the ceramics into two main groups: G1 of 830 °C and G2 of 950 °C. Four other groups with only a few samples each (G3, G4, G5, and G6), and a couple of isolated cases. Group G1, with a distinct peak at 830 °C, consists of eight sherds all of them from the Late Period, Sanagasta style. G2, with a distinct peak at

Table 4
Ceramic groups as determined by maximum temperatures, archaeological cases, and cultural adscription of sherds.

Main groups defined by maximum temperatures, archaeological cases for each group ($n = 30$), and cultural adscription of the sherds					
G1 − 830 °C ($n = 8$)	G2 − 950 °C ($n = 10$)	G3 − 850 °C ($n = 2$)	G4 − 870 °C ($n = 4$)	G5 − 890 °C ($n = 3$)	G6 − 930 °C ($n = 3$)
KLR-7573 (LP)	KLR-7574 (LP)	KLR-7604 (I)	KLR-7577 (EP)	KLR-7601 (I)	KLR-7586 (I)
KLR-7575 (LP)	KLR-7580 (EP)	KLR-7621 (I/DC)	KLR-7602 (I)	KLR-7609 (LP)	KLR-7633 (LP)
KLR-7576 (LP)	KLR-7600 (LP)		KLR-7607 (I)	KLR-7632 (LP)	KLR-7638 (LP)
KLR-7619 (LP)	KLR-7603 (I)		KLR-7635 (LP)		
KLR-7612 (LP)	KLR-7584 (I)				
KLR-7613 (LP)	KLR-7585 (I)				
KLR-7636 (LP)	KLR-7587 (I)				
KLR-7639 (LP)	KLR-7614 (LP)				
	KLR-7634 (LP)				
	KLR-7637 (LP)				

Isolated cases: KLR-7608 (I) (770 °C), KLR-7606 (I/DC) (790 °C), KLR-7611 (LP) (810 °C), KLR-7695 (I) (910 °C), KLR-7615 (LP) (1030 °C), KLR-7579 (EP) undetermined.
References: LP (Late Period), I (Inca), EP (Early Period), I/DC (Inca/Diaguita Chileno).

950 °C, is the largest group consisting of 10 sherds: four Inca sherds, five Sanagasta sherds, and one Saujil sherd. G3 (850 °C) consist by two Inca sherds, one Inca Provincial and one Inca/Diaguita Chileno. G4 (870 °C) holds two Inca sherds, one Sanagasta, and one Saujil. G5 (890 °C) consists of one Inca sherd and two Late Period sherds. G6 (930 °C) holds one Inca sherd and two Late Period Sanagasta style sherds. The isolated cases are 3 sherds of lower firing temperatures: one Inca sherd (770 °C), one Inca/Diaguita Chileno sherd (790 °C), and one Late Period Sanagasta style sherd (810 °C). Then two sherds with higher firing temperatures: one Inca sherd (910 °C) and one Late Period Sanagasta style sherd (1030 °C).

While there is a no systematic separation between Late and Inca period sherds according to the determined maximum firing temperatures, it is obvious that Late Period sherds in general exhibit lower maximum firing temperatures than the Inca sherds (for similar results see Hayashida et al., 2003b). Inca sherds, except for two cases, have maximum firing temperatures above 850 °C.

There is also a clear difference between the Inca Provincial sherds and the two Inca/Diaguita Chileno sherds, the latter exhibiting firing temperatures lower than or equal to 850 °C. The differences observed in the maximum firing temperatures between Late and Inca period sherds are probably related to changes in the firing procedures and kiln technologies which occurred with the appearance of the Incas in the region, which led to a new organization of the whole pottery production. In general there is observed higher and more homogenous maximum firing temperatures of the Inca sherds, which is in accordance with the use of kilns and a better control of temperature and firing atmosphere. Late Period sherds appear to have been fired in conditions exhibiting larger variations in temperatures, probably due to the use of insulated bonfires and even open fires. Perhaps some of the Late Period vessels could have been fired using the new firing procedures and kiln technology brought to the region with the Incas, since it is known that Incas used local labourers to intensify the production of pottery (Hayashida et al., 2003b: 162); however, safe conclusions in this respect requires further investigation. In one particular case a pottery kiln was excavated near Costa de Reyes N° 5 site, where there was found wood used for fuel dating from the Late Period. Whether this was due to the use of old wood, possibly reused timber, or the kiln was indeed in operation in the Late Period remains to be seen.

Therefore the use of the decomposition or neoformation of mineral phases detected by measurements of magnetic susceptibility has proven a powerful tool in revealing archaeological information about the changing firing procedures utilized by the pre-Hispanic societies in the Andean area through time. In the present context, we believe that our data from the Late Period and Inca ceramics exhibit the potential of the method to determine firing temperatures for a broad range of samples with a so far unprecedented accuracy.

5. Conclusions

A new methodology for determining the maximum firing temperature of ceramics and other burnt clay materials using stepwise re-firings and measurements of the magnetic susceptibility has been presented. The method has been validated for two series of clay samples fired at 400, 500, 600, 700, 800, 900, and 1000 °C. The correlation coefficient between the known firing temperature and the experimentally determined firing temperatures was found to be $R = 0.99$ ($R^2 = 0.98$), which is a very satisfactory agreement. The uncertainty in estimating the firing temperature in this validation experiment is determined to be ±25.8 °C (1 sigma). The mineralogical changes responsible for the sudden change in magnetic susceptibility after re-firing exceeding the original firing temperature in the validation series samples are probably the neoformation of Fe-gehlenite and possibly hematite, as shown by powder X-ray diffraction measurements. The method has been demonstrated to work on ceramic samples from very diverse geological clay sources from Denmark, Luristan, Israel, and Argentina. The method has been applied to a set of samples from two cultures which likely had different firing technologies, the Late (*ca.* AD 900–AD 1450) and the Inca (*ca.* AD 1480–AD 1532) periods in the Catamarca region in Northern Argentina. The determined maximum firing temperatures exhibit a two-peak distribution that can be understood in relation to the archaeological context.

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Appendix. Supplementary material

Supplementary data related to this article can be found online at doi:10.1016/j.jas.2012.01.008.

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