



# Metallurgical traditions under Inka rule: a technological study of metals and technical ceramics from the Aconcagua Valley, Central Chile

María Teresa Plaza<sup>\*</sup>, Marcos Martín-Torres

UCL Institute of Archaeology, 31–34 Gordon Square, London WC1H 0PY, United Kingdom



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## ABSTRACT

The spread of the Inka state in the Aconcagua Valley (Central Chile) is thought to have been culturally mediated, avoiding military coercion, and thus leading to different forms of cultural acceptance, resistance or hybridisation. However, there has been no previous attempt to investigate the extent to which these interactions are reflected in the use of metals and metallurgical technologies. Here we present analytical work on metallic artefacts and technical ceramics from Cerro La Cruz and Los Nogales, two Valley sites with evidence dated to the Late Period (ca. AD 1400–1540). The analyses included SEM–EDS, optical microscopy, petrography, XRD and FTIR. The results suggest that the sites represent different technological traditions. At Cerro La Cruz, the style of the metal objects and the lack of tin bronzes reflect continuity with an ancient metallurgical tradition with bases in the Diaguita Culture, rather than a wholesale adoption of an Inka metallurgical tradition. In Los Nogales, the presence of tin bronze and the use of perforated crucibles and other technical ceramics lined with bone ash is consistent with a tradition closely related to the Inka expansion and north-western Argentina, perhaps reflecting a stronger receptivity towards the new technologies. This disparity supports the idea that the Inka domination in the Valley was not forceful, and suggests a closer relationship between the state and some local groups, not previously identified.

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## 1. Introduction

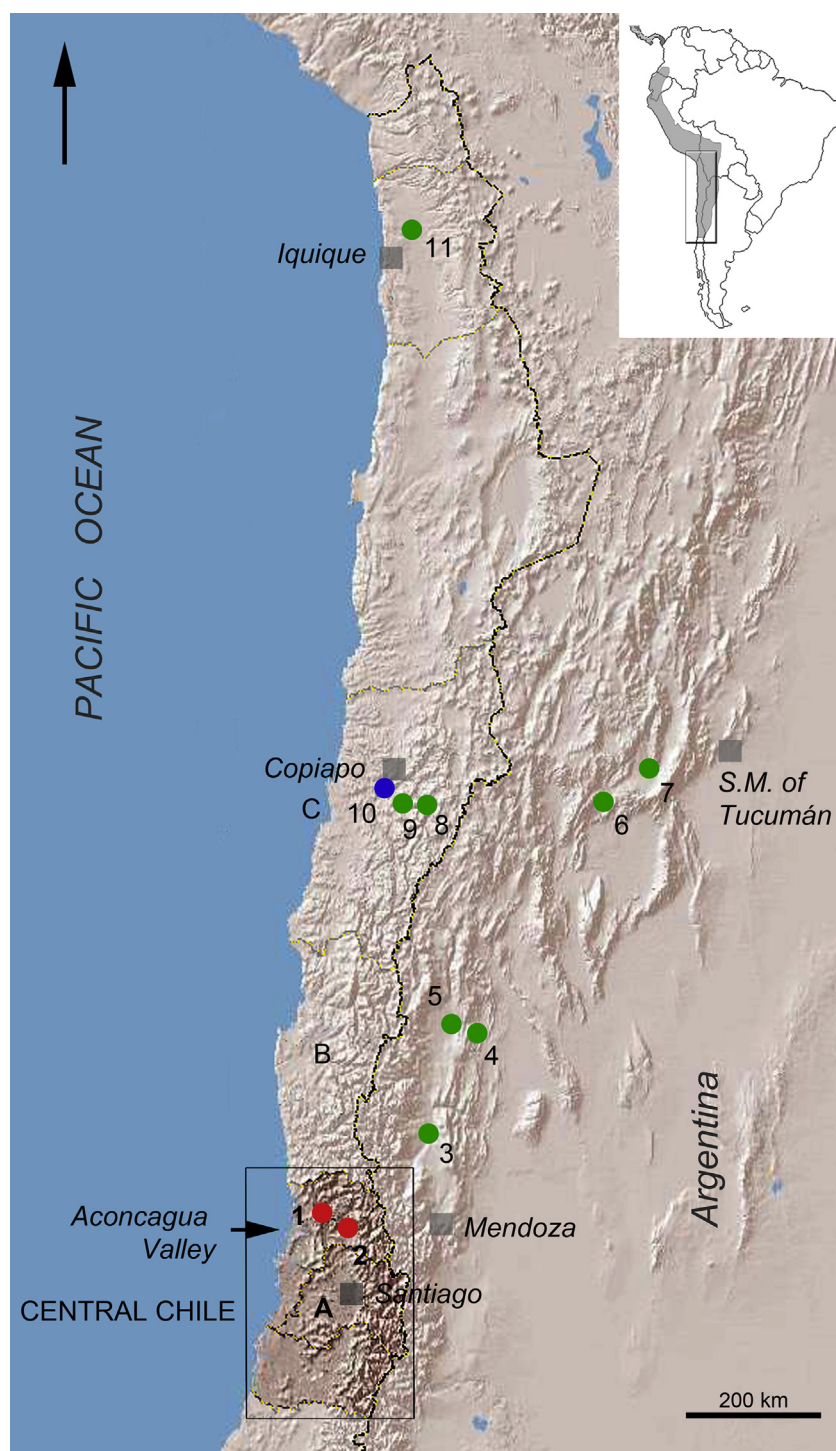
The Aconcagua Valley (or “AV”) is located in the north section of Central Chile, at the southernmost limit of the *Tawantinsuyu* or Inka State. In this area, indigenous communities coexisted with neighbouring groups such as the Diaguitas from the semi-arid North, the Aconcaguas from the Maipo-Mapocho Valley (South) and trans-Andean communities (Fig. 1) (Pavlovic et al., 2012; Sánchez, 2001–2002, 2004; Sánchez et al., 2004). During the Late Intermediate Period (AD 900–1400), no metal artefacts are registered in the AV and all the metallurgical evidence concentrates in the surrounding areas: small scale metallurgical production in the Aconcagua Culture (South) and an established metallurgical tradition in the Diaguita Culture (North) (Campbell and Latorre, 2003; Latorre, 2009; Latorre and López, 2011; Plaza, 2010). However, with the Inka

domination (AD 1400–1540) important changes are noticeable in the AV, including the appearance of more than 60 metallic artefacts, mostly copper-based. Metallurgical remains also appear but at a small scale, suggesting that most metallic artefacts were brought from elsewhere (Plaza, 2010).

The Inka occupation of the AV is characterised by a segregation at settlement level, whereby inkanised communities appear not to interact with the indigenous groups; instead, they establish their sites in new locations, not previously used by native communities (Sánchez, 2004; Pavlovic et al., 2012). Part of the Inka occupation of the AV was mediated by inkanised Diaguita groups (also known as ‘Diaguita-Inka’) who settled in the area integrating the territory to the *Tawantinsuyu*. Evidence of their influence is clear in pottery and burial patterns (González, and Rodríguez, 1993; Sánchez, 2004; Sanhueza, 2001; Vázquez, 1994). Most of the metallic artefacts so far recovered the AV up to date show a Diaguita-Inka style too (Latorre and López, 2011:321), even though some original shapes suggest that some of the artefacts may have been made in the Valley (Plaza, 2010). Interestingly, no classic Inka objects, such as *tumis*, *tupus*, *liwis* or star-shaped maces, have been recorded

<sup>\*</sup> Corresponding author.

E-mail addresses: [maria.plaza.12@ucl.ac.uk](mailto:maria.plaza.12@ucl.ac.uk) (M.T. Plaza), [m.martinon-torres@ucl.ac.uk](mailto:m.martinon-torres@ucl.ac.uk) (M. Martín-Torres).



**Fig. 1.** Map showing the location of the Aconcagua Valley and the sites analysed here: 1- Cerro La Cruz, 2- Los Nogales. Sites with perforated crucibles: 3- Barrealito, 4- Pachimoco, 5- Angualasto, 6- Quillay Wayras, 7- Rincón Chico, 8- Carrizalillo Grande, 9- Iglesia Colorada and 11- Tarapacá Viejo. 10- Viña del Cerro (blue), metallurgical centre. Areas mentioned in the text: A- Maipo-Mapocho Valley, dominated by the Aconcagua Culture; B- Semi-arid North, characterised by the Diaguita Culture; C- Coast of Atacama. Inset, map of South America showing the extension of the Inka territory (shaded area). (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

(González, 2004). The distribution of the finds shows almost all of them in newly founded Inka sites with Diaguita-Inka presence, rather than indigenous sites, suggesting that the use of metal was restricted and marked a difference between native and “inkanised” groups (Plaza, 2010).

Little is known about the metallurgy of the AV during this period, e.g. its technical features and the metals used; whether the

*Tawantinsuyu* introduced tin bronze in the Valley or how metallic objects relate to the few production remains registered. Similarly, we do not know the extent to which metals and metallurgical techniques may reveal different forms of acceptance, rejection or interaction with Inka ideas.

Seeking to pave the way for a better understanding of the metallurgical traditions in the area during the Inka domination, this

**Table 1**

Thermoluminescence and radiocarbon dates published from Cerro La Cruz and Los Nogales, respectively. Cultural periods: Late Intermediate Period-ca. AD 900–1400; Late Period-ca. AD 1400–1540. \*AD calibration based on a 95.4% confidence interval.

Sample	Material	Years BP	AD calibration
<b>Cerro La Cruz – Thermoluminescence dates</b> (Rodríguez et al., 1993)			
UCTL 264	Ceramic sherd black/red on white.	600 ± 70	1390
UCTL 263	Ceramic sherd black/red on white.	560 ± 50	1430
UCTL 261	Ceramic sherd black on white.	560 ± 60	1430
<b>Los Nogales – Radiocarbon dates</b> (Pavlovic, 2010)			
UGAMS 5982	Charcoal	620 ± 25	1336–1398*
UGAMS 5976	Human bone	600 ± 20	1300–1368*
UGAMS 5979	Camelid bone	430 ± 25	1427–1488*
UGAMS 8278	Camelid bone	340 ± 30	1470–1640*

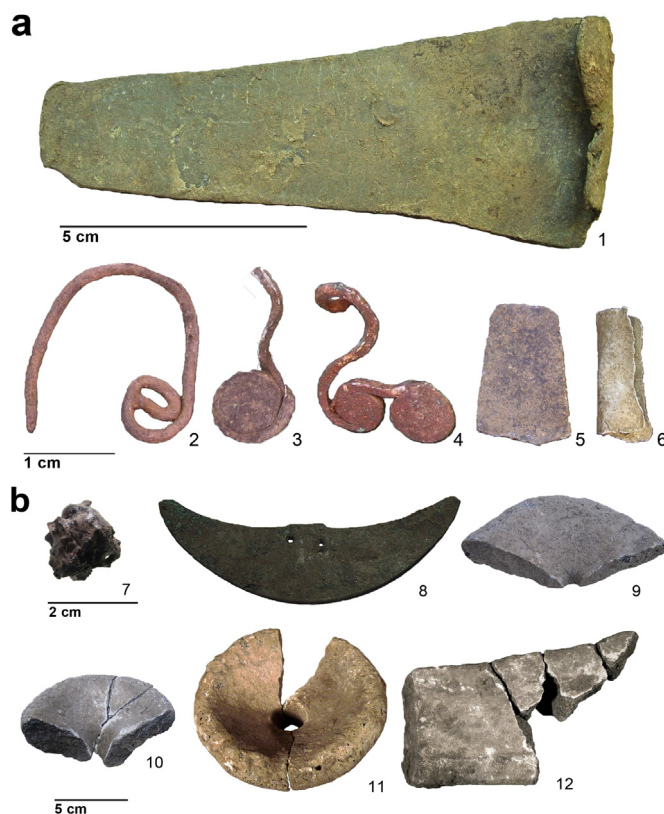
study focuses on the characterisation of a set of production remains and metallic artefacts from two sites. Although significant as the first archaeometallurgical approach to the region, the assemblages are very small and thus any conclusions will have to remain provisional. However, it is shown here that the model of study proposed here has potential and may be adopted in future research. If more sites are brought to the picture in further comparative studies, we may be able to map different metallurgical traditions in the region.

The two sites from the Late Period (AD 1400–1540) of Inka influence considered are Cerro La Cruz (or “CLC”) and Los Nogales (or “LN”) (Fig. 1). CLC is a ritual Inka site with no previous occupations. Thermoluminescence dates place this site at the beginning of the Late Period, AD 1390–1430 (Rodríguez et al., 1993:212). The site has yielded the largest amount of metallic objects in the Valley ( $n = 44$ ); including ornaments and tools, of which 35 were copper-based and 9 were silver-based. It also contains local-Inka, Diaguita-Inka, Aconcagua Culture and indigenous pottery (González, 2011). LN is an indigenous domestic site and burial. Radiocarbon dates indicate a prolonged occupation from the Late Intermediate and into the Late Period (despite the long chronological bracket, no historical materials were identified) (Pavlovic, 2010). LN is associated to indigenous and Aconcagua Culture pottery (Dávila, 2010); no evidence of Inka materials has previously been detected. However, the presence of two metallic artefacts (a semilunar plate and a pair of tweezers) and eleven fragments of technical ceramics, the largest concentration of production remains found in Central Chile up to date, suggests an external influence (Table 1, Fig. 2).

## 2. Methods

The analysis of fourteen samples is reported here: eight metallic objects from CLC and four technical ceramics, one lump of melted waste (possible slag) and one metallic artefact from LN (Fig. 2). Cross-sections of all artefacts were mounted in epoxy resin blocks, polished down to 1 µm and examined using a Leica DM-LM optical microscope.

To further investigate microstructures and determine chemical composition, the resin blocks were carbon-coated and analysed using a Philips XL30 scanning electron microscope with an Oxford Instruments energy dispersive spectrometer, operating at 20 kV and a working distance of 10 mm. Results are an average of five measurements applied in areas of  $200 \times 157 \mu\text{m}$  and  $67 \times 51 \mu\text{m}$  for copper and silver artefacts, respectively; of  $1200 \times 926 \mu\text{m}$  for ceramics and  $600 \times 474 \mu\text{m}$  for the melted waste. Based on the analysis of reference materials, limits of detection (LOD) were established at 0.1 wt%. Chemical data for ceramic and melted waste are presented as stoichiometric oxides in weight percent (wt%), whereas results for metals show all elements as detected. To correct results from corrosion, porosity and beam fluctuations, they were normalised to 100 wt%, but original analytical totals are shown.



**Fig. 2.** Part of the material assemblage of a) Cerro La Cruz, 1–6 and b) Los Nogales, 7–12. All the artefacts showed here were analysed for this study, except N° 11. 1- trapezoidal plate (sample M5), 2–4- earrings (samples M1, M2 and M3), 5–6- fragment of a sheet (sample M6 and M9), 7- melted waste, 8- semilunar plate (M7), 9–10- perforated crucibles (samples R3 and R4), 11- perforated crucible (not analysed), 12- mould (sample R2).

Metallographic observations were made etching the metallic samples using ammonia and hydrogen peroxide 3% for 10–30 s (M5, M6, M8 and M9); aqueous ferric chloride for 3 s (M1 and M7) and nitric acid for 20–30 s (M2, M3 and M4).

Ceramic fabrics of two crucibles and two moulds were also characterised using thin-section petrography, on a Leica DM-EP polarising light microscope. Description of these was based on the system proposed by Quinn (2013:80). The mineral composition of the technical ceramics and their white surface linings were determined using X-ray powder diffraction (XRD). An XPRT-PRO diffractometer was used, with a Spinner PW3064 sample stage. A  $\text{CoK}\alpha$  radiation was applied ( $\lambda = 1.78901$ ) at 40 kV and 30 mA. Diffractograms were recorded in the scanning ranges of  $2\theta$  from  $5.0^\circ$  to  $99.9^\circ$  with a step size of  $0.0170^\circ$ . The results of the white coating presented here were transformed using Bragg's law ( $\lambda = 2d\sin\theta$ ) to fit in the  $\text{CuK}\alpha$  ( $\lambda = 1.541838$ ) parameters and facilitate comparisons with published data.

The nature and firing temperature of the white coating on the technical ceramics were also investigated using Fourier Transform Infrared Spectroscopy (FTIR). Pressed pellets were made of a mixture of powdered sample with KBr. The spectra were collected using a Perkin Elmer 2000 Fourier Transform Infrared Spectrometer. Data were collected in transmission mode applying 30 readings, over a wave length range of  $4000\text{--}370 \text{ cm}^{-1}$ ; with a precision of  $4 \text{ cm}^{-1}$  at  $1 \text{ cm}^{-1}$  intervals. Readings of the atmospheric  $\text{CO}_2$  and  $\text{H}_2\text{O}$  were applied to correct for atmospheric absorption.

All the analyses were carried out at the Wolfson Archaeological Science Laboratory at the UCL Institute of Archaeology, except for



the XRD analyses that were performed in the X-Ray Diffraction Laboratory at the UCL Department of Earth Sciences.

### 3. Results

#### 3.1. Metallic artefacts and melted waste

Within an assemblage of 44 objects from CLC; four earrings, three sheets and one trapezoidal plate were analysed. Their shapes and styles are very similar to those produced by Diaguita metallurgists from the semi-arid North (Latorre, 2009; Latorre and López, 2011; Plaza, 2010:98). However, some of them show particularities, such as the use of double spirals in earrings, which appear to be a local variation of the Diaguita earrings (Fig. 2a:4). Of the copper-based metallic objects from CLC analysed for this project, three were made of pure copper (M1, M2, M4); M3 and M6 contain minor levels of tin (0.6%) and silver (0.6%), respectively; and M5 contains 1.2% arsenic and 1.4% silver (Table 2).

The analyses of their microstructures revealed very similar forming patterns among the small artefacts (M1, M2, M3, M4, M6) (Fig. 3a). In general, all of these present equiaxial grains with a few straight and bent slip lines and twin bands, indicative of artefacts made by cycles of cold-work, annealing and a final step of mild cold-work. However, there is some variation within the group, e.g. the grains of M3 are more elongated suggesting a final step of harder cold-work; on the contrary, the absence of slip lines in M1 indicates that this earring was left in annealed condition. The differences suggest that the five artefacts were made following the same technological sequence, but that working conditions were variable.

The microstructure of the trapezoidal-plate M5 (Fig. 3b) shows small and elongated grains at the edge of the sample and bigger and equiaxial grains at the centre of the piece, the latter with some slip lines. The forming sequence would have included heavy and long working cycles, followed by careful episodes of annealing and further cold-work. The sequence would result in a grain refinement, expanding the surface of contact between grains and incrementing hardness (Scott, 2012:50).

The two silver sheet fragments from CLC analysed were shown to constitute artificial alloys with 2.8 and 9.7% Cu, respectively. Their microstructure shows evidence of heavy and repeated working–annealing cycles, such as the small equiaxial grains in M8 and multiple slip lines and twins bands in M9 (Fig. 3c). The different compositions and microstructures indicate they belonged to different objects.

The compositional data broadly agree with the results of previous XRF analyses of the other objects from the site (F. Garrido pers. comm. 2013), although the latter also revealed the presence of some chisels and thin sheets of arsenical copper, as well as a lead sheet. Overall, the most significant aspects of these datasets are: a) the predominance of pure or very weakly alloyed copper, b) the presence of silver, and c) the near absence of tin – only detected as an impurity in two objects (M3 above, and another sheet with 0.1% Sn reported by F. Garrido pers. comm. 2013).

Only one of the two metallic objects from LN could be analysed, and this turned out to be a high-tin bronze with 10.9% tin and 0.5% sulphur. The metallography of this semi-lunar plate M7 revealed well-formed equiaxial grains with multiple and marked straight and bent slip lines and twin bands (Fig. 3d). The manufacture sequence would have involved cycles of heavy cold-work and full annealing using temperatures high enough to recrystallise the grains, avoid the formation of other phases (that would cause brittleness), but not removing all the strain lines (Scott, 1991:7–9, 2012:50). The bent slip lines and twin bands suggest that a final step of cold-working was applied (Rovira and Gómez, 2003:79).

A small, amorphous lump of suspected slag, black and dark red in colour and 1.5 mm in diameter, was recovered from LN (Fig. 2b:7). The analyses showed it to be a heterogeneous silicate containing shattered grains of quartz and partly molten minerals in addition to newly formed crystals, which indicates exposure to temperatures in excess of 1100 °C. Abundant in this matrix are many small globules (<60 µm, mostly 2 µm) of ferruginous metallic copper and iron–copper sulphides. Although the relatively low bulk iron content and small size of this melted waste make it unlikely that it constitutes a by product of copper smelting, the presence of copper and the evidence of high temperature are coherent with a metallurgical reaction, perhaps related to remelting or refining of copper in a crucible.

#### 3.2. Technical ceramics

Five crucibles and twelve pieces of moulds were found in LN, all of them covered by a thin white coating on both internal and external surfaces (Fig. 2b:9–12). Morphologically, the crucibles are vessels of 39–64 mm height with straight, opening walls and a round base with a hole in the centre. Rim and base diameters are variable, with ranges of 86–119 mm and 20–25 mm, respectively. Maximum capacities range between 80 and 104 cm<sup>3</sup>. Moulds are brick-shaped, with flat surfaces and a rounded base. Their dimensions are 39–43 mm height and 78–94 mm width. Based on the shapes carved on the surfaces for casting, we can reconstruct at least a flat-trapezoidal object. Their fabric is soft and brittle. As detailed below, no traces of heavy non-ferrous metals were recorded in any of these artefacts, thus there is no clear indication that these were ever used for metallurgical processes.

##### 3.2.1. Composition, raw materials and provenance

Petrographic analyses indicate the presence of two related fabric groups, one for moulds (R1–R2) and another for crucibles (R3–R4). Both fabric types contain large amounts of inclusions (50–45%) and meso-to mega voids with some bloating vesicles (15–20%), held together by a relatively small amount of matrix (35%).

In both groups, the main temper material is medium to coarse sand-sized igneous rock fragments (<2 mm, mode = 0.6 mm) of plutonic and volcanic formation, and their main minerals such as plagioclase, hornblende and ferruginous minerals. Quartz, pyroxene, mica, orthoclase feldspar and epidote are also present. Possible parent rocks are microdiorite, andesite or some basalts. These inclusions of igneous rocks from different origins are moderately-sorted, with sub-angular to rounded shapes suggesting their use as tempers probably originating in alluvial deposits. The geological map of the area, showing alluvial sediments formed by weathered rocks of basalt, andesite, dacite and granodiorite, indicates that this temper material could have been obtained locally (SERNAGEOMÍN, 2003).

The main difference between both fabric groups is the bone ash inclusions used as temper in the moulds, and absent in crucibles (Fig. 4). These inclusions are well-sorted, angular in shape and show a smaller range-size distribution compared to the igneous inclusions (<0.6 mm, mode = 0.3 mm), suggesting that the bone ash was crushed and probably sieved before being added to the fabric.

XRD analyses agree with the petrographic data. All samples contained quartz, plagioclase and gehlenite; hydroxylapatite was only detected in moulds. High temperature minerals were also identified: enstatite, diopside, magnetite, haematite, hercynite and cristobalite (Table 3). The clay minerals in the moulds were identified as montmorillonite, a mineral from the smectite family, formed by alteration of basic rocks or volcanic ash (Rice, 2005[1987]). No clay minerals were found in the crucibles, but spinel is present

**Table 2**

Bulk chemical composition of the metallic artefacts by SEM–EDS. Figures are an average of five measurement in areas of  $200 \times 157 \mu\text{m}$  avoiding corrosion and voids.

Bulk chemical composition by SEM–EDS (wt%)								
Id. number	Artefact	O	S	Cu	As	Ag	Sn	Total <sup>a</sup>
M1	Simple earring	<1	nd	100.0	nd	nd	nd	97.2
M2	Spiral	<1	nd	100.0	nd	nd	nd	97.1
M3	Double spiral	<1	nd	99.4	nd	nd	0.6	104.1
M4	Wire	<1	nd	100.0	nd	nd	nd	99.3
M5	Trapezoidal plate	<1	nd	97.4	1.2	1.4	nd	99.8
M6	Fragment of sheet	<1	nd	99.4	nd	0.6	nd	101.5
M7	Semilunar plate	<1	0.5	88.6	nd	nd	10.9	104.9
M8	Fragment of sheet	1.1	nd	9.7	nd	89.9	nd	101.4
M9	Fragment of sheet	<1	nd	2.8	nd	96.8	nd	100.0

<sup>a</sup> Totals presented here are analytical totals, before normalisation. Oxygen was present in most measurements below 1% indicating the low incidence of corrosion, figures over 1% were specified. “nd”: not detected.

instead, which may represent a high temperature form of montmorillonite (Rice, 2005:92 [1987]).

An approximation to the bulk composition of the ceramic fabrics was obtained by averaging SEM–EDS scans of relatively large areas (Table 4a). While silica and alumina are the main oxides in the crucibles, lime and phosphate levels in the moulds exceed those of alumina, which is consistent with the presence of bone temper. Also significant are the relatively high levels of soda (2.7–4.2%) and magnesia (1.2–2.1%). The high levels of soda may be explained by the intermedia to basic nature of the fabric inclusions with abundant Na-rich plagioclase feldspars, such as albite, oligoclase, andesine and anorthite, as identified by XRD. This analysis also registered enstatite, diopside and Mg-rich spinel, probable cause of the high levels of magnesia. No traces of metallic or heavy elements was detected during examination of the technical ceramics.

We attempted to analyse the ceramic matrix by SEM–EDS spot analyses that avoided large inclusions. However, the limited amount of matrix registered among the abundant temper made this task difficult, and the results for the moulds still reflect the enrichment in lime and phosphate likely related to the bone temper (Table 4b). In order to facilitate the comparison between the clays employed for crucibles and moulds, we neglected the contribution from the bone composition in the latter and re-normalised the SEM–EDS results, thus obtaining a rough approximation of the composition of the mould clays without the bone temper (adapted from Martín-Torres et al., 2009, who used it to determine the additives mixed with bone ash in metallurgical cupels). Here, we assumed that all the  $\text{P}_2\text{O}_5$  in the fabric derived from bone, and based on the average  $\text{CaO}/\text{P}_2\text{O}_5$  ratio measured for the bone inclusions (1.3), we calculated the amount of CaO in the fabrics that derived from the bone (Table 4c). We subsequently removed the calculated ‘bone CaO’ and all the  $\text{P}_2\text{O}_5$  from the data before re-normalising the results.

The re-normalised data (Table 4d) shows that the clay used in the manufacture of the ceramics was non-calcareous in all cases, and the compositional similarity between moulds and crucibles makes it plausible that they derive from the same clay. A plot of these re-normalised results in a ternary diagram based on clays compositions (Tite et al., 1982:112), confirms that LN ceramics were made of a smectite and illite/smectite clay, supporting XRD data (Fig. 5).

### 3.2.2. Temperatures

Fig. 6 gives the thermal stability ranges of some minerals found by XRD in moulds and crucibles. The presence of montmorillonite in the moulds indicates that these did not exceed 900 °C (Rice,

2005:92 [1987]). Magnetite and diopside start to form at ~820–900 °C, their presence thus indicating temperatures over ~820 °C (Maritan et al., 2005:41). Both crucibles contained spinel, which appears around 900 °C (Rice, 2005:92 [1987]) and gehlenite, enstatite, magnetite, diopside and anorthite, which confirm temperatures over 800 °C. Although cristobalite was found in crucibles and this phase is generally said to form around 1050 °C (Rice, 2005:95 [1987]), experiments have shown that cristobalite can form at temperatures below 1000 °C in a soda-rich environment (Cole, 1935:151,153), as was the case with these ceramics (R3 contained up to 4% of  $\text{Na}_2\text{O}$ ). Based on the XRD data, therefore, the estimated firing temperatures for moulds are between ~820° and 900 °C, and for crucibles above ~900°, possibly up to ~1000–1050 °C. Given the lack of evidence for use residues in any of these, for now we assume that these temperatures reflect the original firing of these ceramics, as opposed to high-temperature exposure during subsequent use.

### 3.2.3. The coating

All the technical ceramics were covered in- and outside by a white layer around 800  $\mu\text{m}$  thick, made of angular grains with a spongy, cellular texture and <200  $\mu\text{m}$  in size (Fig. 7). SEM–EDS area analyses of the coating detected 86–97% of calcium phosphate, with low levels of silica, alumina, iron oxide, magnesia, chlorine, soda and potash. Spot analyses on the porous grains revealed ~99% of calcium phosphate, with a  $\text{CaO}/\text{P}_2\text{O}_5$  ratio of 1.3 (Table 5).

The composition and the texture of the inclusions strongly indicate that they constitute fragments of ground bone ash. The main mineral component of bone is hydroxylapatite (HAp)  $\text{Ca}_5(\text{PO}_4)_3(\text{OH})$ , a calcium phosphate mineral that may also contain calcium carbonate, fluoride, magnesium, sodium, potassium and traces of chlorine and iron (Martín-Torres et al., 2008, 2009; Rivera-Muñoz, 2011). The low concentrations of alumina, silica and iron oxides, indicate that pure bone was used to prepare the coating, ruling out a mixture with other materials (Martín-Torres et al., 2009; White, 2010).

XRD patterns for the archaeological samples are highly consistent with reference patterns for apatite, however these do not allow for an easy discrimination between a geological or biogenic origin. FTIR was used therefore, to distinguish between both (Odriozola and Matinez-Blanes, 2007). The FTIR spectra show the characteristic absorption bands of the functional groups distinctive to HAp (Fig. 8; Table 6). When compared to the spectra of experimental calcined bones and a geological apatite, LN specimens match the patterns of the calcined bones in almost all the absorption bands (Fig. 9). Additionally, all spectra showed a sharp peak at  $630 \text{ cm}^{-1}$  known to be correlated with the loss of the absorbed water, which develops at temperatures over 650 °C and disappears around 900 °C. Its presence confirms the burnt-bone nature of the coating, while suggesting the use of temperatures below 900 °C (Berzina-Cimdina and Borodajenko, 2012; Koutsopoulos, 2002; Odriozola and Hurtado, 2005, 2007; Odriozola and Martinez-Blanes, 2007).

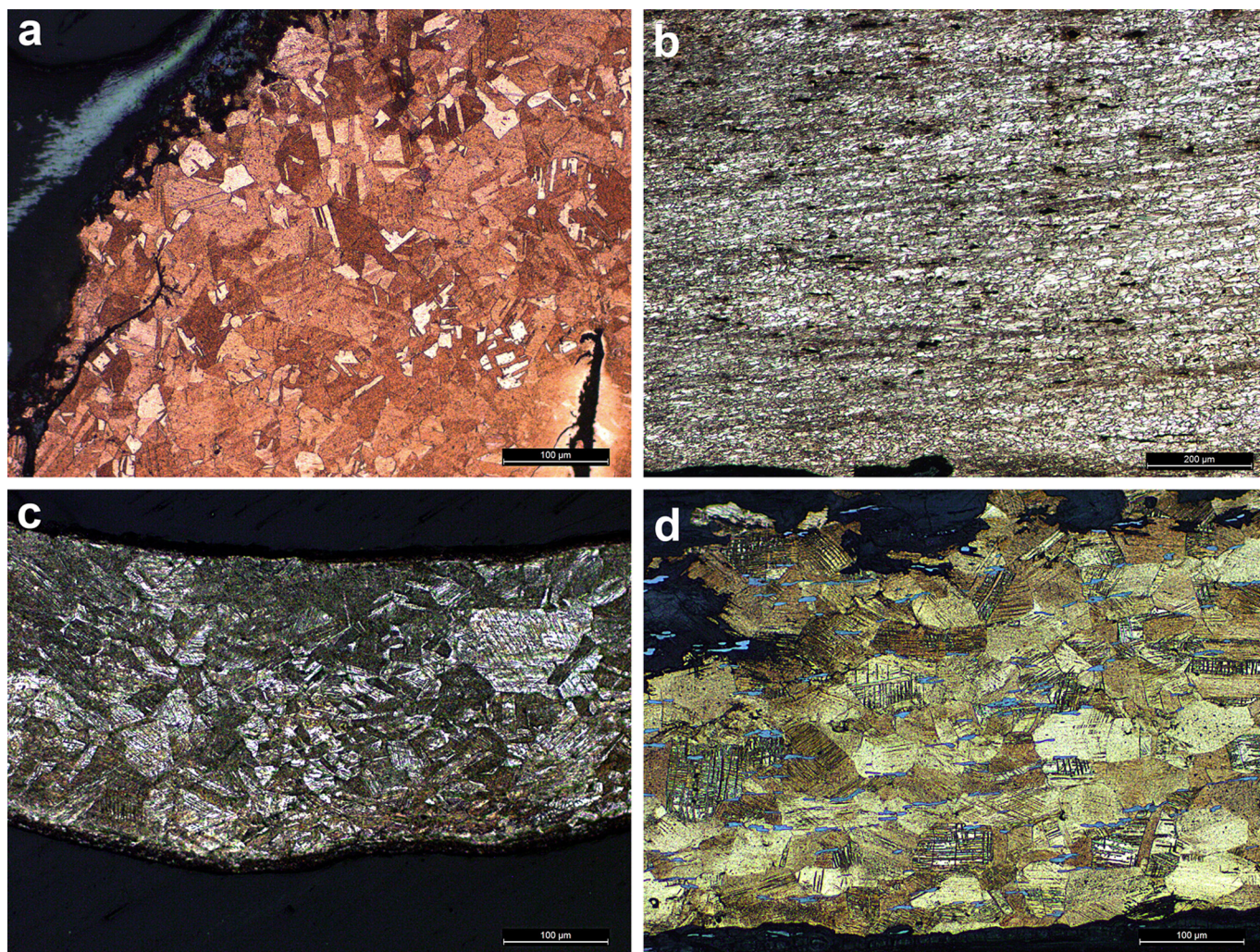
## 4. Discussion

### 4.1. Metallic artefacts

Based on the technological sequences, CLC artefacts made of pure copper, including those with minor percentages of silver and tin, can be considered a coherent technical group (M1, M2, M3, M4, M6), in that they all underwent the same sequence of techniques, even if showing variability in terms of final working steps.

Both plates M5 and M7 were heavily hammered, producing however different microstructures probably based on their different alloys. Sample M5 contained ~1.3% of arsenic. According to





**Fig. 3.** Optical microscopy images under plane polarised light (PPL), all at the same magnification (200 $\times$ , 596  $\mu$ m width). a) Copper earring M1, note the slip lines and twins bands. b) Plate M5, note the grain refinement, compared with the other samples. The direction of work is also indicated by the inclusions of cuprite (corrosion) and banding. c) Silver sheet M9, note the equiaxial grains with twin bands and multiple slip lines. d) Bronze semi-lunar plate M7, note the equiaxial grains and the large amount of slip lines, several of them are bent. The elongated blue inclusions are copper sulphides. (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

Lechtman (1996:481) amounts from 0.5% arsenic produce perceptible changes in the metal properties, increasing its malleability and hardness. The sample analysed was taken from a folded section, and metalworkers probably took advantage of the malleability of this arsenical bronze to fold the plate and increase its hardness. In the case of the high-tin bronze from LN (M7), the high tin content produced a hard piece which required heavy work in order to shape it; it was also subjected to high or long enough annealing temperatures to avoid brittleness, producing a relatively resistant and hard object of a bright yellow colour.

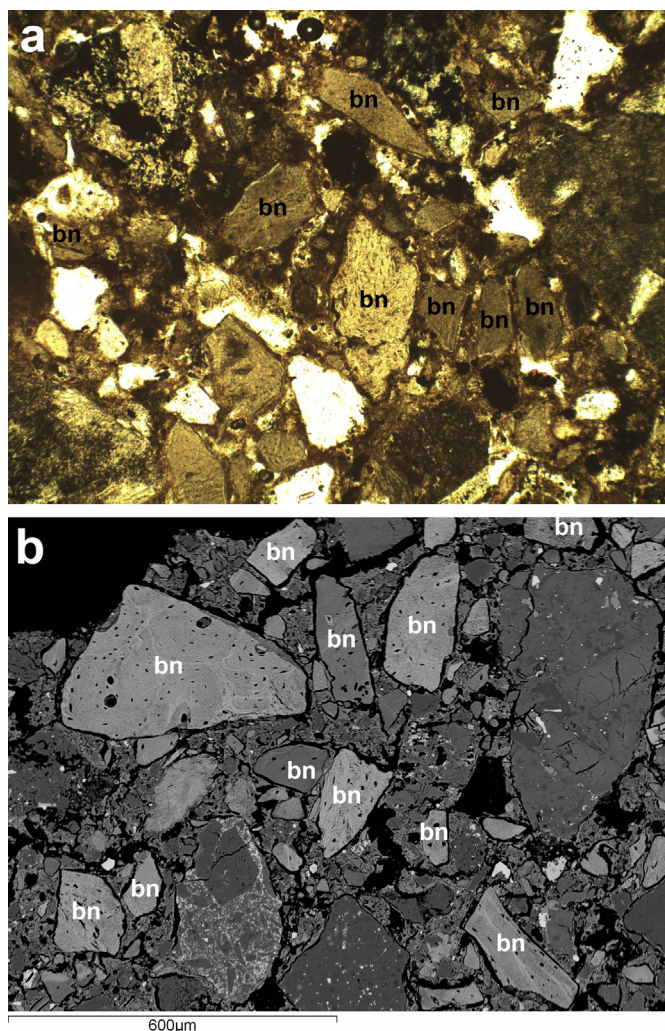
Regarding the silver sheets, the absence of lead above detection limits suggest that cupellation was not used, since this process usually leaves trace levels of 0.2–2% Pb (Gordon and Knopf, 2007:45; Scott, 2012:38). This would rule out the possible use of local ores, which are mixed with lead (Ulriksen, 1990), and is consistent with the use of non-local silver chloride or sulphide ores. However, it should be borne in mind that a refining process to remove the lead in the silver is also possible (Schlosser et al., 2012:2886).

The techniques applied to produce these artefacts suggest that metalworkers knew how to control the degree of work to exploit

the properties of the different metals used (e.g. pure copper or high tin bronze). However, the internal variation within the first group of relatively pure copper objects, would also indicate that working conditions were variable and not standardised.

Of more interest are the different compositions, specifically their minor elements, which indicate the use of a variety of ores and a differential provision of metals in both sites. Mineralogically speaking, central and northern Chile (18–34°S) are rich in large porphyry copper–molybdenum belts, stratabounds of copper–silver, copper–arsenates and copper–sulfarsenides (Lechtman, 2007; Makasev et al., 2004, 2007; Serrano et al., 1996). Based on this, copper objects with silver and/or arsenic impurities such as those found in CLC are common along the Chilean territory, including Central Chile. It is probable that metallurgists recognised these impure ores and selected them intentionally for the manufacture of harder objects, which is also consistent with the identification of arsenical copper chisels by the XRF study (F. Garrido pers. comm. 2013). While the local exploitation of such ores has been proposed in other Chilean regions (e.g. Salazar et al., 2010), no such evidence has been found in Central Chile (Troncoso et al., 2012:313). Instead, evidence for metal extraction is found in the





**Fig. 4.** Microstructure of mould R1, showing the cellular structure of the bone inclusions. a) thin-section image under PPL, width 1.5 mm; b) backscattered electron image.

Diaguita territory to the north, which also contains similar mineral deposits (Cantarutti, 2013; Niemeyer, 1979–81, 1983). This suggests that at least some of the metal found at CLC could have been brought from northern areas as ingots (bars?), scrap or finished objects. The variety of impurities detected would also support the idea of metals from different metal sources coming into the site. Still, the presence of particular designs would be indicative of a local manufacture.

The occurrence of cassiterite ores in South America, on the other hand, is much more localised, concentrated specifically in present-day north-west Argentina (or “NWA”) and Bolivia. At the same time, it is well documented that during the Inka period, tin bronze was traded and exported from sites such as Rincón Chico in NWA (González, 2004:298, 2010). In Chile, use of this alloy is recorded in the coastal communities of the North (Salazar et al., 2010), the Inka metallurgical centre Viña del Cerro (Niemeyer 1986:181 in Moyano, 2009:26) and the coast of Atacama (Gutiérrez, 2012). Thus, it would seem reasonable to expect the presence of tin bronze in CLC, a ritual site with abundant archaeological signs of inkansation.

Against this background, the low incidence of tin in CLC is most remarkable. Considering the pieces analysed here, plus previous analyses on a further six artefacts from the site (Rodríguez et al., 1993:206) and unpublished XRF (F. Garrido pers. comm. 2013), it

**Table 3**

Minerals identified by XRD analysis. Pl- Plagioclase; Ksf- K-feldspars. (\*) List of the minerals that were searched for, but were not found.

Minerals identified in the technological ceramics by XRD		
Mineral	Ideal formula	Samples
Quartz	SiO <sub>2</sub>	R1–R2–R3–R4
Gehlenite	Ca <sub>2</sub> Al(AlSi) <sub>2</sub> O <sub>7</sub>	R1–R2–R3–R4
Calcite	CaCO <sub>3</sub>	R1–R2–R3–R4
Hydroxylapatite (HAp)	Ca <sub>5</sub> (PO <sub>4</sub> ) <sub>3</sub> (OH)	R1–R2
Montmorillonite	(Al,[Na,Mg])(Si <sub>2</sub> O <sub>5</sub> ) <sub>2</sub> (OH)	R1–R2
Andesine (Pl)	(Na,Ca)(Si,Al) <sub>4</sub> O <sub>8</sub>	R1–R2–R4
Enstatite	Mg <sub>2</sub> Si <sub>2</sub> O <sub>6</sub>	R1–R2–R4
Diopside	CaMgSi <sub>2</sub> O <sub>6</sub>	R1–R2–R4
Anorthite (Pl)	CaAl <sub>2</sub> Si <sub>2</sub> O <sub>8</sub>	R1–R3
Magnetite	Fe <sub>3</sub> O <sub>4</sub>	R1–R3–R4
Cristobalite	SiO <sub>2</sub>	R3–R4
Spinel	MgAl <sub>2</sub> O <sub>4</sub>	R3–R4
Oligoclase (Pl)	(Na,Ca)(Si,Al) <sub>4</sub> O <sub>8</sub>	R1
Albite (Pl)	NaAlSi <sub>3</sub> O <sub>8</sub>	R2
Haematite	Fe <sub>2</sub> O <sub>3</sub>	R2
Sanidine (Kfs)	(K,Na)(Si,Al) <sub>4</sub> O <sub>8</sub>	R2
Hercynite	Fe <sub>2</sub> Al <sub>2</sub> O <sub>4</sub>	R4
Sauconite	(NaZn(SiAl) <sub>2</sub> O <sub>5</sub> (OH)(H <sub>2</sub> O) <sub>2</sub>	R4

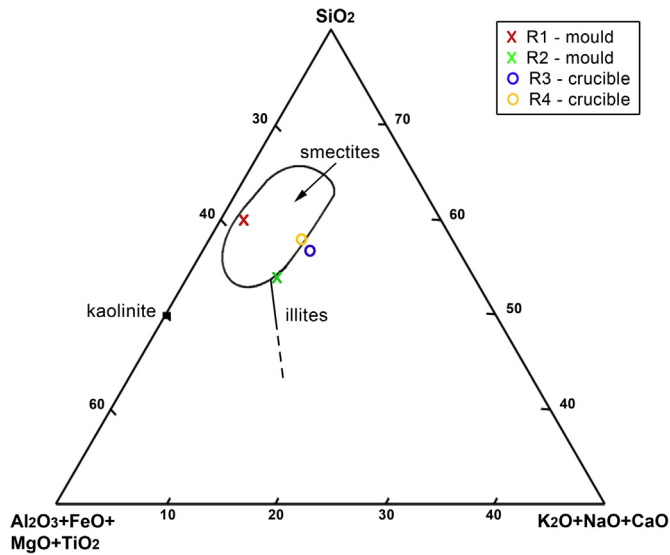
\*Minerals checked: chlorite, glauconite, illite, vermiculite, kaolinite, whitlockite, mullite, tridymite and fayalite.

is noteworthy that just two out of 44 artefacts contain tin, and only in very minor levels. It has been proposed that ore deposits of tin and copper in South America are never mixed, which would indicate that the low percentage of tin in two CLC objects is unlikely to derive from an ore impurity (González, 2004:174,141). In this case, it would be more likely that this low tin is the signature of some scrap bronze recycled and diluted with pure copper, but that such practice was only very sporadic. However, stannite (Cu<sub>2</sub>FeSnS<sub>4</sub>) has been recently identified in mineral deposits from Bolivia, indicating that the possibility of a natural impurity cannot be ruled out (Keutsch and Brodtkorb, 2008). Either way, the bottom line is that CLC had access to foreign materials but they were not receptors of

**Table 4**

Chemical composition of the refractories by SEM–EDS. R1–R2: moulds. R3–R4: crucibles. a) Bulk chemical composition of the ceramic bodies. Results are normalised at 100%. b) Chemical composition of the matrix of the technical ceramics. Point analysis avoiding inclusions and voids. c) Point analysis in bone inclusions of the body in R1; figure is an average of three measurements. Alumina, silica and iron oxide are considered contamination of the surrounded matrix. d) Same figures of “b” re-normalised after subtracting bone composition (CaO and P<sub>2</sub>O<sub>5</sub>).

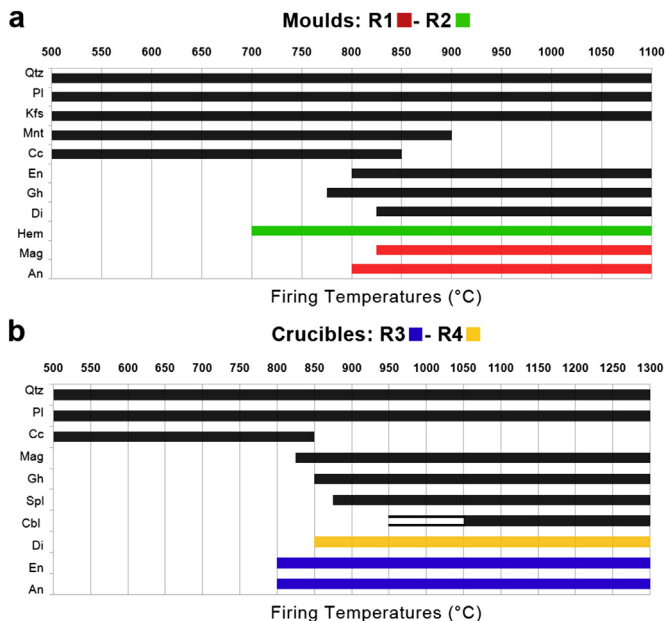
Chemical composition of the refractories by SEM–EDS (wt%)										
	Na <sub>2</sub> O	MgO	Al <sub>2</sub> O <sub>3</sub>	SiO <sub>2</sub>	P <sub>2</sub> O <sub>5</sub>	Cl	K <sub>2</sub> O	CaO	TiO <sub>2</sub>	FeO
<b>a) – Bulk body</b>										
R1	2.7	1.2	11.7	38.9	17.6	0.2	1.2	22.0	0.6	4.0
R2	3.4	1.6	14.9	49.1	10.0	0.2	1.3	13.8	0.6	5.1
R3	3.3	2.1	18.6	57.6	nd	nd	1.8	4.9	1.4	10.2
R4	4.2	2.1	19.1	59.2	nd	0.1	2.0	4.6	1.0	7.8
<b>b) – Matrix</b>										
R1	0.6	3.1	19.4	49.1	8.1	0.1	0.9	10.7	0.5	7.4
R2	1.6	2.6	18.7	41.2	10.5	0.3	1.5	16.8	0.7	6.1
R3	3.4	1.9	17.6	58.0	0.2	nd	1.6	3.8	1.4	11.9
R4	3.9	1.6	23.7	57.1	nd	nd	2.2	4.3	0.5	6.8
<b>c) – Bone inclusions within the body</b>										
R1	nd	0.6	0.6	0.5	41.7	1.3	nd	54.9	nd	0.4
CaO/P <sub>2</sub> O <sub>5</sub> ratio: 1.3										
<b>d) – Matrix data re-normalised</b>										
R1	0.8	3.9	23.8	60.3	–	0.2	1.2	0.2	0.6	9.1
R2	2.1	3.4	24.6	54.4	–	0.4	2.0	4.1	1.0	8.0
R3	3.4	1.9	17.7	58.2	–	nd	1.6	3.6	1.4	12.0
R4	3.9	1.6	23.7	57.1	nd	nd	2.2	4.3	0.5	6.8



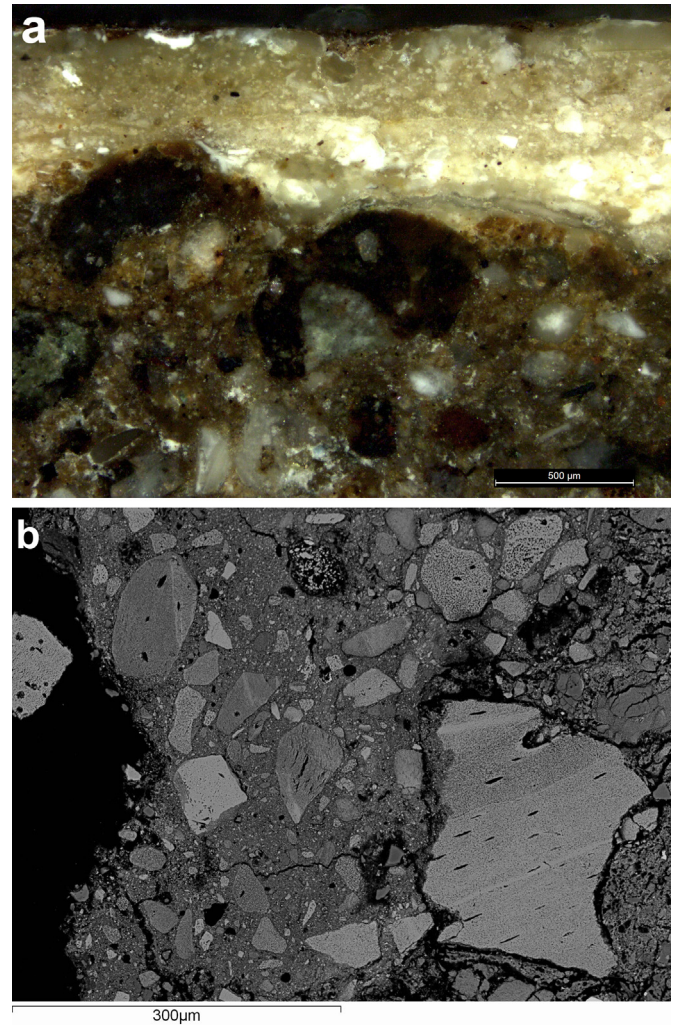
**Fig. 5.** Ternary diagram indicating the composition of the mayor clays minerals and the samples from Los Nogales. Note that chemical composition of the ceramics analysed here plot within smectites and illite/smectite clays (modified from Tite et al., 1982: Fig. 5).

bronze artefacts – contrary to what one would expect in an Inka site (González, 2004; Lechtman, 1978).

Based on the similarity in manufacturing techniques and design of earring M3 with other earrings from CLC, and the recovery in the coast of Atacama of bars (ingots?) with 0.4–0.6% Sn (Gutiérrez, 2012:95), it is reasonable to suggest that M3 was locally made, using copper metal stock that brought tin as an impurity. Due to the low tin content, its workability and final appearance would be very similar to that of the pure copper ornaments.



**Fig. 6.** Thermal behaviour of some minerals detected by XRD. a) moulds and b) crucibles. Black bars indicate minerals found in common; colour bars indicate minerals found in a single sample. Minerals key: **Qtz**-quartz; **Pl**-plagioclase (albite, andesine); **Kfs**-potassium feldspars (sanidine); **Mnt**-montmorillonite; **Cc**-calcite; **En**-enstatite; **Gh**-Gehlenite; **Di**-diopside; **Hem**-haematite; **Mag**-magnetite; **An**-anorthite; **Spl**-spinel; **Cbl**-cristobalite. (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)



**Fig. 7.** a) Optical microscope image showing the white layer in sample R2 under cross-polarised light; this particular layer has an average width of 600 µm (Image width: 2.4 mm). b) Backscattered electron image, detail of a cross section of the white layer in sample R1. Note the angular and irregular bone inclusions of different sizes (Image width: 600 µm).

Overall, the evidence supports the idea that CLC actively chose not to include tin bronze in their metal repertoire, i.e. they were selecting pure copper and the tin impurity was unnoticed and unintended. While one could regard this as the result of an external imposition, when we consider the main features of the site, an active choice on the part of CLC seems more likely. Firstly, the site is clearly Inka based on its architecture, location, function and local-Inka pottery (Acuto et al., 2010), evidencing that the *Tawantinsuyu* intervened the traditional patterns and used its technologies in other fields. It also contains non-local raw materials, such as combarbalite, turquoise, obsidian, malachite and most important, silver; showing access to a variety of materials from other areas (González, 2011:101; Rodríguez et al., 1993:210). Moreover, gold and silver were highly valued within the Inka ideology and were introduced in Central Chile and the semi-arid North by the *Tawantinsuyu* (González, 2004; Latorre, 2009:88); the presence of silver in CLC indicates that elements of the Inka metallurgical tradition were circulating but just a few were adopted.

The case of LN, with a tin-bronze semi-lunar plate, tells a different story. Its style, composition and manufacturing techniques are characteristic of the Inka metallurgical tradition



**Table 5**

Chemical composition of the white coating by SEM–EDS. R1–R2: moulds. R3–R4: crucibles. Calcium and phosphorus percentages are shown in bold.

Chemical composition of the white coating by SEM–EDS (wt%)									
	Na <sub>2</sub> O	MgO	Al <sub>2</sub> O <sub>3</sub>	SiO <sub>2</sub>	P <sub>2</sub> O <sub>5</sub>	Cl	K <sub>2</sub> O	CaO	FeO
<b>Bulk composition</b>									
R1	0.3	0.8	1.5	4.1	<b>39.7</b>	0.5	nd	<b>52.6</b>	0.5
R2	0.5	0.5	1.0	1.7	<b>41.6</b>	0.4	nd	<b>54.2</b>	nd
R3	0.8	1.2	1.9	6.4	<b>37.1</b>	0.4	0.3	<b>49.1</b>	2.8
R4	0.2	0.5	0.3	1.3	<b>41.9</b>	0.9	nd	<b>54.9</b>	nd
Mean	0.5	0.7	1.2	3.4	<b>40.1</b>	0.6	0.1	<b>52.7</b>	0.8
<b>Bone inclusions</b>									
R1	nd	0.3	nd	nd	<b>42.7</b>	0.2	nd	<b>56.8</b>	nd
R2	0.1	0.3	nd	nd	<b>42.7</b>	0.2	nd	<b>56.7</b>	nd
R3	0.2	0.8	nd	nd	<b>43.3</b>	0.3	0.1	<b>55.4</b>	nd
R4	nd	0.2	nd	nd	<b>42.8</b>	0.7	nd	<b>56.3</b>	nd
Mean	0.1	0.4	–	–	<b>42.9</b>	0.3	0.02	<b>56.3</b>	–
<b>Bone cement</b>									
R1	nd	0.4	0.3	0.5	<b>42.6</b>	0.6	nd	<b>55.6</b>	nd
R2	nd	0.5	1.0	0.6	<b>42.1</b>	0.9	nd	<b>54.9</b>	nd
R3	0.1	0.5	0.2	0.4	<b>42.6</b>	0.5	nd	<b>55.7</b>	nd
R4	0.2	0.6	nd	nd	<b>42.7</b>	1.1	nd	<b>55.5</b>	nd
Mean	0.1	0.6	0.4	0.4	<b>42.5</b>	0.7	–	<b>55.4</b>	–

(Lechtman, 1978:504) and starkly different from those found in CLC, making it difficult to technologically link both sites. The forging of high tin bronze required specific techniques that were not used in CLC. Moreover, the ~11% Sn produced a very different piece in appearance (bright yellow); furthermore, considering that the design is unique in the area, it is suggested that this artefact was probably introduced to the region as a finished object. Although the metal assemblage from the site is limited to a mere two artefacts, the one object analysed does indicate that people at LN were more receptive to tin bronze – a characteristically Inka alloy.

#### 4.2. Technical ceramics

The bone ash-lined moulds and especially the crucibles found in LN belong to the same type as those reported in metallurgical sites

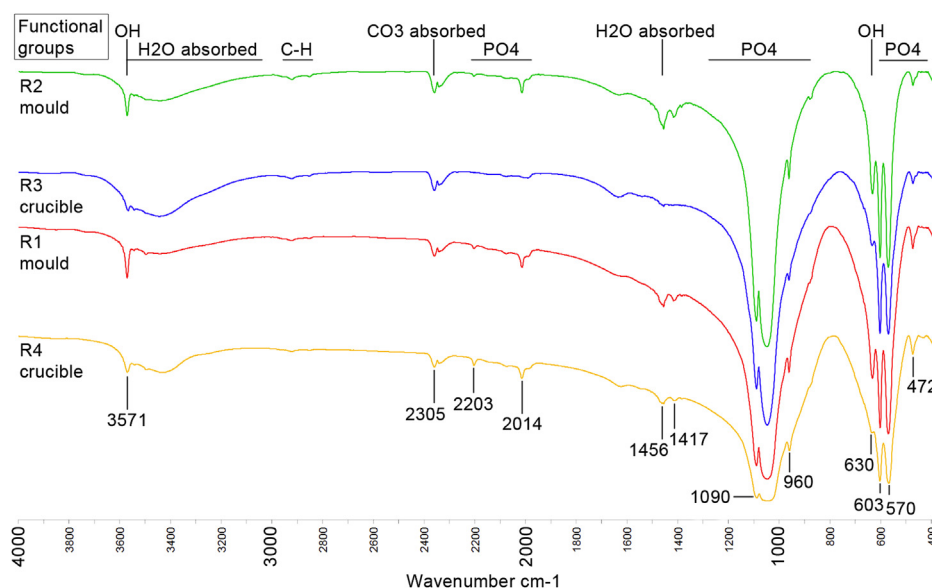
**Table 6**

Description of the main absorption bands in Fig. 8 [explanation of Fig. 8].

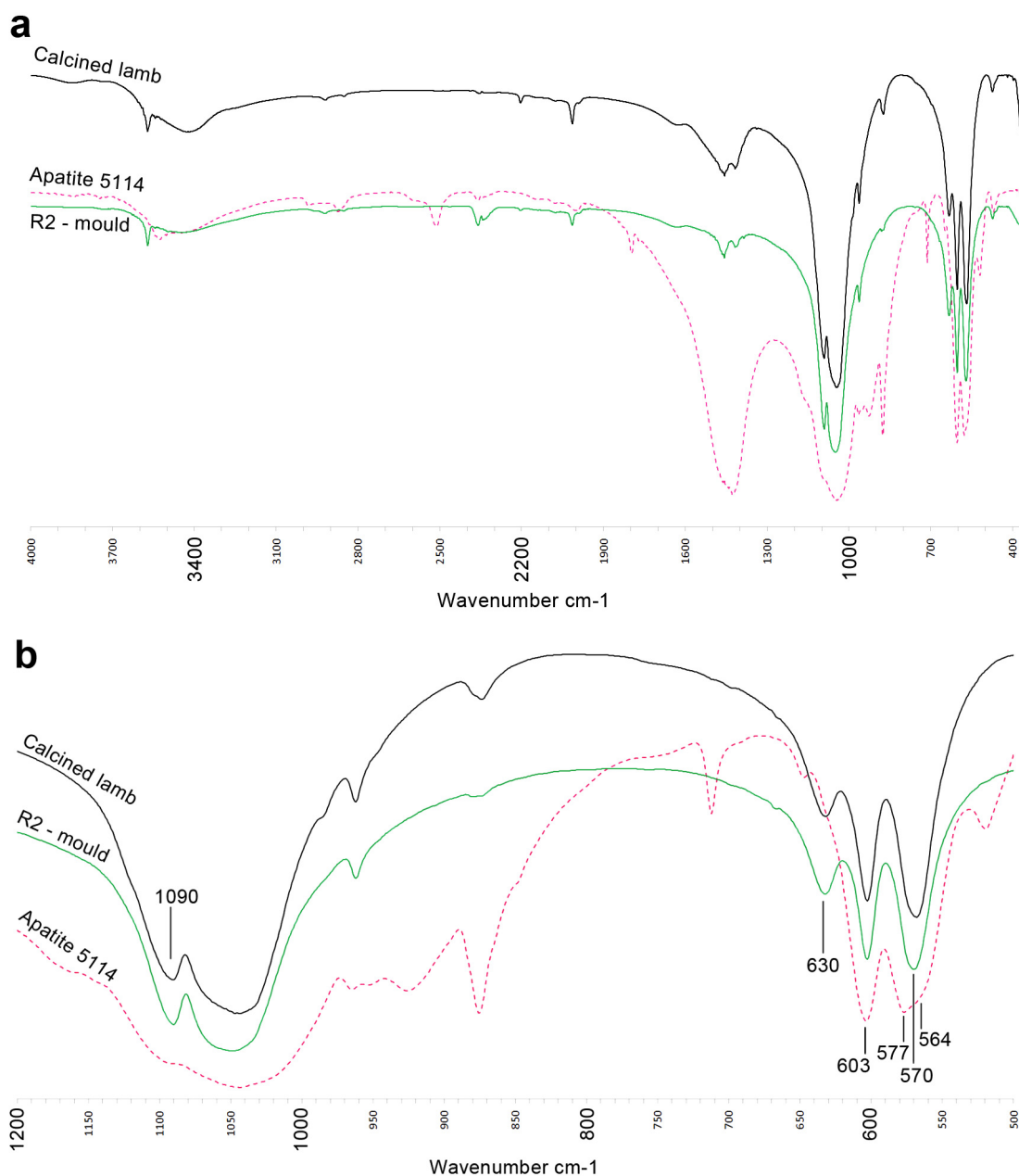
Absorption bands of the functional groups distinctive to HAP		
Peaks (cm <sup>-1</sup> )	Correlations	References
At ~3570 and at 630 (sharp)	Related to the absorbed water. Represent the stretching and librational modes of the hydroxyl group (OH <sup>-</sup> ). Peak 630 develops at high temperatures over 650 °C.	Arsad et al., 2011; Berzina-Cimdina and Borodajenko, 2012; Koutsopoulos, 2002
At ~1090 and 960	Asymmetrical stretching mode. Formed after thermal treatment.	Berzina-Cimdina and Borodajenko, 2012; Odriozola and Hurtado, 2005
At 570 and 603	Double band of asymmetric and symmetric deformation modes.	Odriozola and Hurtado 2005; Koutsopoulos, 2002
At 472	Double degenerated bending mode of the O–P–O bonds.	Arsad et al., 2011; Koutsopoulos, 2002
At 2203 and 2014 (sharp)	Reported as characteristics of biological apatite.	Gluzman et al., 2009
At ~1456 and 1417	Bands absent in geological apatite.	Odriozola and Hurtado, 2005

from NWA and Inka sites from northern Chile (see sites in Fig. 1), thus allowing us to confidently identify them as metallurgical ceramics even if they were not used. Although they occur before the Inka, their geographic spread and frequency increase under the Inka expansion. It has been proposed that this type of perforated crucible was used to collect the molten metal, which would then be poured in different moulds through the base perforation (Niemeyer, 1979–1981). Whatever their specific functioning, their manufacture and use were unquestionably complex (González, 1997; González and Gluzman, 2009; Niemeyer, 1979–1981; Pifferetti, 2004).

Besides the conspicuous base perforation, technical ceramics from LN share two main features with those from other metallurgical sites (Fig. 1:3–11). The first one is the calcareous lining. The use of this layer started before the Inka, during the Aguada phase in



**Fig. 8.** FTIR spectra of the four samples from Los Nogales and their main absorption bands. R1 and R2 are moulds and R3 and R4 are crucibles. Note that all samples show a similar profile [associated to Table 6]. (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)



**Fig. 9.** a) FTIR spectra of the experimental calcined bone, geological apatite and one archaeological sample (R2). Note the similarities between the archaeological and calcined bone samples, compared with the profile of the geological apatite. b) Detail of the finger print region of the spectra. Bands at 1090 and 630  $\text{cm}^{-1}$  are characteristic for burnt bone. According to [Odriozola and Hurtado \(2005\)](#), the peak at  $\sim 577$  with a shoulder at  $564 \text{ cm}^{-1}$  is typical for geological apatite.

NWA (AD 500–900), together with the first use of tin-bronze and the development of a complex casting technology in the area ([González, 1997, 2002a–b, 2004, 2010](#)). The analyses reported here demonstrate that the coating from LN was made of pure, ground, calcined bone. A similar recipe has been identified in Rincón Chico 15 ([González, 2010](#)) and Tarapacá Viejo ([Zori et al., 2012](#)), but variants based on other calcium compounds were used in Carrizalillo Grande ([Niemeyer, 1979–1981](#)) and Quillay Wayra ([Raffino et al., 1996](#)). These different recipes indicate local adaptations to the technological tradition, and a competent knowledge in the use of available materials to produce a comparable product.

The second common feature is the fabric preparation, notably the abundance of coarse rock inclusions mixed with a small portion

of clay ([Campo, 2001; González, 2002a, 2010; Niemeyer, 1979–1981; Pradell et al., 2010](#)). In the case of LN, the smectite-montmorillonite clay was not very refractory, due to its low alumina and high alkali contents ([Rice, 2005:48 \[1987\]](#)). However, the addition of large amounts of relatively coarse sand-sized igneous rocks as temper ([Hein et al., 2007](#)) and the application of the calcareous, refractory layer, would no doubt improve the performance characteristics of the ceramics.

A particular feature found in the moulds of LN is the use of bone ash as temper, which has not been reported elsewhere. The addition of bone ash to clays has some advantages: it prevents cracks and splitting during drying, it improves workability ([Walter et al., 2004](#)) and, as it has a similar thermal expansion coefficient as



ceramics, it produces a strong and relatively flexible vessel (Stilborg, 2001). Moreover, bone is very refractory and chemically inert – it enhances the resistance to high temperatures and does not react with oxidised metals (Martín-Torres et al., 2008, 2009; White, 2010). Since the moulds analysed here were fired at relatively low temperatures and had friable fabrics, the bone ash temper would improve the paste workability before firing, while the limited shrinkage would help preserve the original design shaped on them.

#### 4.3. Metallurgical traditions under Inka rule

Even though the sample analysed is admittedly small, the metallurgical evidence presented above allow us to suggest that, technologically speaking, both sites represent different traditions introduced in the AV during the Inka expansion. On the one hand, the provision of metals at CLC is dominated by relatively pure copper, with some incidence of arsenical copper, based on ores that were relatively common along the Chilean territory. The anecdotal presence of tin-bearing alloys at the site indicates that this community had access to circulating metals including tin bronze, but that they generally chose not to use them. Significantly, the presence of silver artefacts at the site indicates that potentially prestigious, exotic metals were accessible, which reinforces the suggestion that the absence of tin bronzes is the result of deliberate behaviour at the local level. This pattern in the composition of the metals, together with the absence of typologically classic inkansised artefacts, seems consistent with a technological conservatism that allowed an increase in metal use but largely maintained traditional raw materials and techniques different from those spread by Inkas and north-western Argentinean cultures (see González, 2002a, 2004; Lechtman 2007). The marked Diaguita style of most metal artefacts at CLC (Plaza, 2010) would connect this assemblage to an ancient Diaguita metallurgical tradition developed in the semi-arid North since the Animas phase, ca. AD 700 (Latorre, 2009). Bearing the above in mind, plus the presence of some original designs not found in the semi-arid North, we propose that CLC may represent a local metallurgical development in the AV, based on the Diaguita metallurgical tradition and probably maintained by Diaguita-Inka artisans.

On the other hand, the one object from LN analysed represents the work in tin bronze, an imported material with a long tradition in NWA and widely expanded and used by the Inka. The presence of a bronze plate and crucibles with a hole in the base may not be a coincidence, as these technical ceramics are usually related to bronze production (Debenedetti, 1917; González, 2010; Niemeyer 1986 in Moyano, 2009:26; Raffino et al., 1996). While the seemingly unused metallurgical ceramics and the small lump of copper-rich melted waste are not enough to suggest bronze production at the site, the metallurgical evidence indicates that people from LN may have been participating in different networks connecting sites from central- and north-western Argentina, the Atacama region and northern Chile (Fig. 1). Significantly, these regions are considered the mining and metallurgical core from the Southern Andes, specifically driven and linked by the *Tawantinsuyu* (Raffino et al., 1996:65). Even if LN is located in the boundaries of this core, the evidence places the site more strongly in the metallurgical sphere that would become idiosyncratic of the Inkas, as opposed to more local developments aligned with the Diaguita tradition.

It is widely accepted that Inka conquest in Central Chile did not use military coercion; instead, it appears the phenomenon was based on the diffusion and transmission of Inka ideology, resulting in different forms of acceptance or rejection among the local populations, and thus stimulating variable levels of integration to the *Tawantinsuyu* (Pavlovic et al., 2012:566). Against this background,

continuity and/or changes in technological traditions can be expected to reveal the nature of these relationships, acting as indicators of acceptance, cultural resistance and/or strategies applied by the Inka to generate a new order (González and Tarragó, 2004).

The technical conservatism proposed for the metals at CLC could thus reflect a form of cultural resistance in the metallurgical arena, which could be explained by the strong roots in a well established metallurgical tradition from the semi-arid North. Despite the inkansisation clearly manifest in the pottery and architecture of CLC, the Inka materiality was not completely adopted by this group, as evidenced in the rejection and/or modification of certain aspects of the dominant technology, such as the use of bronze, gold and the manufacture or use of classic Inka designs in metal (González and Tarragó, 2004). A different situation is observed in LN, an indigenous site without any other Diaguita-Inka or Inka-local evidence. In this case, tin bronze and the metallurgical equipment may represent the introduction of a new technological tradition in the area spread by the *Tawantinsuyu* (González, 1997). People from LN thus appear more receptive to the ideas about metals brought by the *Tawantinsuyu* adopting not just the use of bronze artefacts, but also a new type of metallurgical ceramics to which some innovations were applied, such as the use of bone temper. The lack of an indigenous metallurgical tradition during the previous period was probably key in facilitating the almost wholesale adoption of the Inka tradition.

The identification of two metallurgical traditions introduced in the AV during the Inka expansion agrees with the thesis that the Inka domain in this area was culturally mediated, revealing a variety of ways in which the *Tawantinsuyu* approached the groups settled there; the conservatism of CLC, showing some particularities but maintaining a Diaguita technological base, is also consistent with a non-coercive scheme (Pavlovic et al., 2012; Troncoso et al., 2012). At the same time, the evidence of LN suggests that despite the fact that direct Inka evidence in the AV is usually not found at indigenous sites, Inka ideas and materials circulated, and some were adopted by native groups.

## 5. Conclusions

Technological analyses have allowed us to propose the existence of at least two metallurgical traditions introduced in the AV during the Late Period: the first in Cerro La Cruz, based on a Diaguita substrate with an ancient metallurgical tradition; and the second in Los Nogales, an indigenous site adopting the technologies related to NWA and northern Chile that would become characteristic of the Inka.

CLC is characterised by the use of relative pure copper, with some incidence of arsenical copper and silver, subjected to a series of cold-work and annealing sequences, sometimes showing a final event of cold-work. The ores exploited are likely to have been secondary copper minerals with small amounts of arsenic and silver, which occur abundantly across the Chilean territory. The object designs share traits with the traditional Diaguita style, and typical Inka typologies are absent.

The metal assemblage from LN is much smaller but shows the presence of high-tin bronze. The bronze piece analysed was heavily worked, followed by long annealing episodes and a final step of cold-work. The design, new in the region, and the use of bronze suggest that the artefact, or its prototype, was imported. Additionally, the technical ceramics found in LN correspond to metallurgical equipment described in other Inka sites from northern Chile and NWA. While coarse-grained, perforated crucibles with a calcareous lining are reported from other sites, the purity of the bone-ash lining and the use of bone temper for the moulds are

characteristic of LN, suggesting some flexibility and independence in the manufacture of these materials.

Both groups of materials denote the existence of different types of specialised metallurgical knowledge at each site. More importantly, these differences may also reveal different attitudes towards the Inka domination. At CLC, the Inka state did not interfere in the metallurgical tradition developed from a technological Diaguita base; with the only possible exception of silver, no exogenous metals or techniques were introduced, suggesting perhaps cultural resistance expressed in the metallurgical sphere. At LN, where Inka presence is more limited but there was no prior indigenous metallurgical tradition, people were more receptive to the new materials and methods: the presence of bronze and perforated crucibles may represent a first attempt by the state to introduce their metallurgical technology in the area through specific communities different from those of Diaguita background.

As we mentioned, these suggestions are based on the analysis of relatively small assemblages and two sites only, so they may change in the future. For now, the metallurgical evidence is consistent with the proposal that the Inka implemented a variety of strategies to incorporate the local communities into the state; both maintaining some traditions and introducing new technologies in the same area.

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## References

- Acuto, F., Troncoso, A., Ferrari, A., Pavlovic, D., Jacob, C., Gilardenghi, E., Sánchez, R., Amuendo, C., Smith, M., 2010. Espacialidad incaica en los andes del sur: La colonización simbólica del paisaje y la ritualidad Inca en Chile Central y el Valle Calchaquí. *Actas del XVII Congr. Nac. Arqueol. Argent. Mendoza* 3 (26), 1297–1302.
- Arsad, M., Lee, P., Hung, L., 2011. Synthesis and Characterization of Hydroxyapatite Nanoparticles and  $\beta$ -TCP Particles. In: 2nd International Conference on Biotechnology and Food Science, vol. 7, pp. 184–188. Singapore.
- Berzina-Cimdina, L., Borodajenko, N., 2012. Chapter 6: Research of calcium phosphates using fourier transform infrared spectroscopy. In: Theophanides, T. (Ed.), *Infrared Spectrometry. Materials Science Engineering and Technology*. Intech, Rijeka, pp. 123–148.
- Campbell, R., Latorre, E., 2003. Rescatando una materialidad olvidada: Síntesis, problemática y perspectivas en torno al trabajo prehispánico de metales de Chile Central. *Bol. la Soc. Chil. Arqueol.* 35/36, 47–61.
- Campo, P., 2001. Interpretando los fragmentos de refractarios del poblado prehispánico de Rincón Chico, Catamarca. *Actas XIV Congr. Arqueol. Argent.* 9, 15–24.
- Cantarutti, G., 2013. Mining under the Inca rule in North-Central Chile: the los Infieles mining complex. In: Tripcevich, N., Vaughn, K. (Eds.), *Mining and Quarrying in the Ancient Andes. Sociopolitical, Economic and Symbolic Dimensions*. Springer, New York, pp. 185–212.
- Cole, S., 1935. The conversion of quartz into cristobalite below 1000°C, and some properties of the cristobalite formed. *J. Am. Ceram. Soc.* 18 (1–12), 149–154.
- Dávila, C., 2010. Análisis cerámico del sitio Los Nogales. Comuna de San Esteban, V región. Unpublished report.
- Debenedetti, S., 1917. Investigaciones arqueológicas en los Valles Preandinos de la provincia de San Juan. *Rev. la Univ. B. Aires* 15, 122–167.
- Gluzman, G., González, L., Martín-Torres, M., Odrizola, C., 2009. Technical ceramics and metallurgical secrets: the case of prehispanic Rincón Chico workshop (NW Argentina). In: Poster session presented at: Proceedings 10th European Meeting on Ancient Ceramics, September 2009. London.
- González, A., 2011. Reevaluación del sitio Cerro La Cruz su función en las estrategias de dominio incaico en el curso medio del Aconcagua (Unpublished BA thesis). Universidad de Chile.
- González, C., Rodríguez, A., 1993. Análisis de las prácticas funerarias incaicas de Chile Central. *Actas del XII Congr. Nac. Arqueol. Chil.* 2, 223–234. Temuco.
- González, L., 1997. Cuerpos ardientes. Interacción Surandina y tecnología metalúrgica. *Estud. Atacameños* 14, 175–188.
- González, L., 2002a. Heredará el bronce. Incas y metalúrgica en el sur del valle de Yocavil. *Intersecc. Antropol.* 3, 55–68.
- González, L., 2002b. A sangre y fuego. Nuevos datos sobre la metalurgia Aguada. *Estud. Atacameños* 24, 21–37.
- González, L., 2004. Bronces Sin Nombre. La Metalurgia Prehispánica en el Noroeste Argentino, first ed. Fundación CEPPA, Buenos Aires.
- González, L., 2010. Fuegos sagrados. El taller metalúrgico del Sitio 15 de Rincón Chico (Catamarca, Argentina). *Bol. del Mus. Chil. Arte Precolomb.* 15 (1), 47–62.
- González, L., Gluzman, G., 2009. Agárrame si puedes. Métodos de sujeción de crisoles en el taller metalúrgico prehispánico del sitio 15 de Rincón Chico. *Anu. Arqueol.* 1, 139–152.
- González, L., Tarragó, M., 2004. Dominación, resistencia y tecnología: la ocupación incaica en el Noroeste Argentino. *Chungara* 36 (2), 393–406.
- Gordon, R., Knopf, R., 2007. Late horizon silver, copper and tin from Machu Picchu. *J. Archaeol. Sci.* 34, 38–47.
- Gutiérrez, C., 2012. El alma de los metales: Producción tecnológica de piezas metálicas en el litoral de la Región de Atacama (Unpublished BA thesis). Universidad de Chile.
- Hein, A., Kilikoglou, V., Kassianidou, V., 2007. Chemical and mineralogical examination of metallurgical ceramics from a Late Bronze Age copper smelting site in Cyprus. *J. Archaeol. Sci.* 34, 141–154.
- Keutsch, F., Brodtkorb, M.D., 2008. Metalliferous paragenesis of the San José mine, Oruro, Bolivia. *J. South Am. Earth Sci.* 25 (4), 485–491.
- Koutsopoulos, S., 2002. Synthesis and characterization of hydroxylapatite crystals: a review study on the analytical methods. *J. Biomed. Mater. Res.* 62 (4), 600–612.
- Latorre, E., 2009. De adornos y herramientas nacidos del fuego: una caracterización del trabajo de metales en la cultura Diaguita (c.a. 900–1536 d.C.) (Unpublished BA thesis). Universidad de Chile.
- Latorre, E., López, P., 2011. Los metales de la cultura Diaguita Chilena (Ca. 900–1536 DC): una aproximación metodológica e interpretativa. *Intersecc. Antropol.* 12, 319–332.
- Lechtman, H., 1978. Temas de Metalurgia Andina. In: Ravines, R. (Ed.), *Tecnología Andina*. Instituto de Estudios Peruanos, Lima.
- Lechtman, H., 1996. Arsenic bronze: dirty copper or chosen Alloy? a view from the Americas. *J. Field Archaeol.* 23 (4), 477–514.
- Lechtman, H., 2007. The Inka, and Andean metallurgical tradition. In: Burger, R., Morris, C., Matos, R. (Eds.), *Variations in the Expression of Inka Power. A Symposium at Dumbarton Oaks, October 1997*. Dumbarton Oaks Research Library and Collection, Washington D.C., pp. 313–355.
- Makasev, V., Munizaga, F., McWilliams, M., Fanning, M., Marthur, R., Ruiz, J., Zentilli, M., 2004. New chronology for El Teniente, Chilean Andes, from U-Pb, 40Ar/39Ar, Re-Os and fission-track dating. Implications for the evolution of a supergiant porphyry Cu-Mo deposit. *Soc. Econ. Geol. Special Publ.* 11, 15–54.
- Makasev, V., Townley, B., Palacios, C., Camus, F., 2007. Metallic ore deposits. In: Moreno, T., Gibbons, W. (Eds.), *The Geology of Chile*. The geological society, London.
- Maritan, L., Mazzoli, C., Noradi, L., Russo, U., 2005. Second Iron Age grey pottery from Este (northeastern Italy): study of provenance and technology. *Appl. Clay Sci.* 29, 31–44.
- Martín-Torres, M., Thomas, N., Rehren, Th., Mongiatti, A., 2008. Some problems and potentials of the study of cupellation remains: the case of post-medieval Montbeliard, France. *ArcheoSciences* 32, 59–70.
- Martín-Torres, M., Rehren, Th., Thomas, N., Mongiatti, A., 2009. Identifying materials, recipes and choices: some suggestions for the study of archaeological cupels. In: *Archaeometallurgy in Europe*. AIM Associazione Italiana di Metallurgia, Milan, pp. 435–445.
- Moyano, R., 2009. Sol, luna y huacas en el valle de Copiapó. El Ushnu de Viña del Cerro como lugar de observación astronómica y culto a las montañas en Atacama (Unpublished BA thesis). University of Chile.
- Niemeyer, H., 1979–1981. Dos tipos de crisoles prehispánicos del Norte Chico, Chile. *Bol. del Mus. Arqueol. la Serena* 17, 92–109.
- Niemeyer, H., 1983. Viña del Cerro, expresión metalúrgica inca en el valle de Copiapó. *Creces* 4, 32–35.
- Odrizola, C., Hurtado, V., 2005. Tecnología y producción de decoraciones cerámicas campaniformes con relleno de hueso en la cuenca media del Guadiana. Av. *Arqueometría*. *Actas VI Congr. Ibérico Arqueometría* 71–79 (Girona).
- Odrizola, C., Hurtado, V., 2007. The manufacturing process of 3rd millennium BC bone based incised pottery decoration from the Middle Guadiana river basin (Badajoz, Spain). *J. Archaeol. Sci.* 34 (11), 1794–1803.
- Odrizola, C., Matinez-Blanes, J.M., 2007. Estimate of firing temperatures through bone based chalcolithic decorated pottery. *J. Therm. Anal. Calorim.* 87 (1), 135–141.
- Pavlovic, D., 2010. Informe preliminar salvataje sitio arqueológico Los Nogales, comuna de San Esteban, provincia de Los Andes, V región de Valparaíso (Unpublished report).
- Pavlovic, D., Troncoso, A., Sánchez, R., Pascual, D., 2012. A tiger in the valley: roads, architecture and Inca rituality in the upper course of Aconcagua valley. *Chungara* 44, 551–569.



- Pifferetti, A., 2004. Las cucharas de colada por el fondo en la metalurgia prehispánica. In: *Actas del Congreso CONAMET/SAM La Serena, Chile*.
- Plaza, M.T., 2010. Estudio sobre la metalurgia Incaica en Chile Central durante el Periodo Alfarero Tardío (Unpublished BA thesis). Universidad de Chile.
- Pradell, T., González, L., Gluzman, G., 2010. Estudios técnicos de materiales refractarios del noroeste Argentino. *La arqueometría Argent. Latinoamerica* 85–90.
- Quinn, P., 2013. *Ceramic Petrography*. Archaeopress, Oxford.
- Raffino, R., Iturriza, R., Iacona, A., Capparelli, A., Gobbo, D., Montes, V., Vázquez, R., 1996. Quillay: centro metalúrgico Inka en el noroeste argentino. *Tawantinsuyu* 2, 59–68.
- Rice, P., 2005. [1987] *Pottery Analysis. A Sourcebook*. University of Chicago Press, Chicago.
- Rivera-Muñoz, E., 2011. Hydroxyapatite based materials: synthesis and characterization. In: Fazel-Rezai, R. (Ed.), *Biomedical Engineering: Frontiers and Challenges*. Intech, Rijeka, pp. 75–98.
- Rodríguez, A., Morales, R., González, C., Jackson, D., 1993. Cerro La Cruz: Un enclave económico administrativo incaico, curso medio del Aconcagua (Chile Central). *Actas del XII Congr. Nac. Arqueol. Chil.* 2, 201–221 (Temuco).
- Rovira, S., Gómez, P., 2003. Las primeras etapas metalúrgicas en la Península Ibérica. III Estudios metalográficos. Instituto Universitario Ortega y Gasset, Madrid.
- Sánchez, R., 2001–2002. El Tawantinsuyu Salvaje en el Finis Terrae Australis (Chile Central). *Rev. Chil. Antropol.* 16, 87–127.
- Sánchez, R., 2004. El Tawantinsuyu en Aconcagua (Chile Central). *Chungara* 36 (2), 325–336.
- Sánchez, R., Pavlovic, D., González, P., Troncoso, A., 2004. Curso superior del río Aconcagua. Un área de interdigitación cultural Periodos Intermedio Tardío y Tardío. *Chungara* 36 (2), 753–766.
- Sanhueza, L., 2001. El aribalo Inka en Chile Central. *Werkén* 2, 47–69.
- Salazar, D., Figueroa, V., Mille, B., Morata, D., Salinas, H., 2010. Metalurgia prehispánica en las sociedades costeras del norte de Chile (Quebrada Mamilla, Tocopilla). *Estud. Atacameños* 40, 23–42.
- Schlosser, S., Reinecke, A., Schwab, R., Pernicka, E., Sonetra, S., Laychour, V., 2012. Early Cambodian gold and silver from Prohear: composition, trace elements and gilding. *J. Archaeol. Sci.* 39, 2877–2887.
- Scott, D., 1991. *Metallography and Microstructure of Ancient and Historic Metals*. J. Paul Getty Press, Los Angeles.
- Scott, D., 2012. *Ancient Metals: Microstructure and Metallurgy In: Copper and Copper Alloys*, Second ed., vol. I. Conservation Science Press.
- Serrano, L., Vargas, R., Stambuck, V., Aguilar, C., Galeb, M., Holmgren, C., Contreras, A., Godoy, S., Vela, I., Skewes, M., Stern, C., 1996. The late miocene to early pliocene Río Blanco-Los Bronces copper deposit, Central Chilean Andes. In: Camus, F., Sillitoe, R., Petersen, R. (Eds.), *Andean Copper Deposits. New Discoveries, Mineralisations, Styles and Metallogeny*, Society of economic geologists, Special publication 5, pp. 119–130.
- SERNAGEOMIN (Servicio Nacional de Geología y Minería), 2003. Mapa geológico de Chile: versión digital. *Publicación Geológica Digital*, 4. <http://www.ipgp.fr/~dechabal/Geol-millon.pdf>.
- Stilborg, O., 2001. Temper for the sake of coherence: analyses of bone and chaff tempered ceramics from Iron Age Scandinavia. *Eur. J. Archaeol.* 4 (3), 398–404.
- Tite, M., Freestone, I., Meeks, D., Bimson, M., 1982. The use of scanning electron microscopy in the technological examination of ancient ceramics. In: Olin, J., Franklin, A. (Eds.), *Archaeological Ceramics*. Smithsonian Institution Press, Washington, D.C., pp. 109–120.
- Troncoso, A., Pavlovic, D., Acuto, F., Sánchez, R., González-García, C., 2012. Complejo arquitectónico Cerro Mercachas: arquitectura y ritualidad incaica en Chile Central. *Rev. Española la Antropol. Am.* 42 (2), 293–319.
- Ulriksen, C., 1990. Mapa metalogénico de Chile entre los 18° y 34° S (1: 1.000.000). *Bol. del SERNAGEOMIN* 42.
- Vázquez, M., 1994. Contextos cerámicos incaicos de Chile Central. In: *Actas del 2° taller de Arqueología de Chile Central*. Santiago.
- Walter, T., Paine, R., Horni, H., 2004. Histological examination of bone-tempered pottery from mission Espíritu Santo (41VT11), Victoria County, Texas. *J. Archaeol. Sci.* 31, 393–398.
- White, H., 2010. Legge's Mount, the Tower of London, London. Scientific Examination of the Cupels. Technology Report. London. In: *Research Department Report Series N° 57-2010*. English Heritage.
- Zori, C., Tropper, P., Scott, D., 2012. Copper production in late prehispanic northern Chile. *J. Archaeol. Sci.* 40 (2), 1165–1175.