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ARCHAEOMETRY AT THE ARGENTINE NATIONAL ATOMIC ENERGY COMMISSION: CHARACTERIZATION OF ARGENTINE NORTHWESTERN POTTERY

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This paper provides a description of the Nuclear Analytical Techniques Group of the Argentine National Atomic Energy Commission, at the Ezeiza Atomic Center. In 1963, this laboratory performed the first application of neutron activation analysis in Argentina and currently employs the technique to analyse a variety of matrices, including archaeological ceramics. The group has collaborated on different research projects since the 1980s, when the first characterization of archaeological ceramics was performed. A brief history of the laboratory and the results from the analysis of potsherds and raw materials from northwestern Argentina are presented below.

KEYWORDS: INAA, ARCHAEOLOGICAL CERAMICS, NORTHWESTERN ARGENTINA

INTRODUCTION

The Nuclear Analytical Techniques (NAT) Group of the Radiochemistry Unit at the Ezeiza Atomic Center is one of the two neutron activation analysis (NAA) laboratories under the Argentine National Atomic Energy Commission (CNEA). NAT is a small research group comprised of six researchers, mostly dedicated to the development of NAA applications in different fields, but NAT also performs services for other national and provincial institutions and laboratories and for private customers. The irradiations are currently performed at the RA-3 reactor of the Ezeiza Atomic Center, although if necessary, two other reactors can be used: the RA-1 reactor (40 kW), at the Constituyentes Atomic Center and the RA-6 (500 kW), at the Bariloche Atomic Center. The RA-3 reactor is a pool-type reactor with a 8.5 MW power output and a thermal flux of 6×10^{13} cm^{-2} s^{-1} that operates on 20% enriched uranium fuel, moderated and refrigerated by water. The reactor was inaugurated in December 1967 and went critical the following year at 3 MW. Over the years, RA-3 has undergone several upgrades, not only due to technological advances in instrumentation but also to meet various production and research needs. During the early years, a pneumatic (P-tube) irradiation facility had been in use, but it was shut down due to problems with the system. Today, tests for two new pneumatic irradiation tubes for short-lived radioisotopes are well advanced and a new P-tube facility is expected to be completed and operating by the end of 2007.

The NAT Group obtained accreditation from the Argentine national accreditation body (OAA) in 2001 for the determination of Se by radiochemical NAA in biological samples, and for the analysis of Co, Fe, La, Sc, Sm and Th in geological and related matrices by instrumental NAA. The laboratory was re-accredited in 2004 (Resnizky *et al*. 2006).

Financial support for the laboratory is supplied by the CNEA, different national and international funded projects, and from service analyses for users outside the institution. The group also collaborates with other departments of the CNEA and with universities and research institutes throughout Argentina and other Latin American countries.

The first application of NAA in Argentina was in 1963 using the CNEA RA-1 reactor, and involved the determination of As in nails and hair for a forensic case. A few other research projects followed until 1969, when the laboratory moved to the Ezeiza Atomic Center to begin work with the then recently inaugurated RA-3 reactor. In 1970, the first radiochemical NAA application was performed, determining Ba, Cs and Rb in biological matrices, and from then on the range of analysed matrices and elements continued to grow. It was not until the mid-1980s that the first archaeological samples were analysed. Since then, the laboratory has collaborated on multiple archaeological research projects involving chemical characterization of ceramics. These collaborations include not only the generation of data, but also the analysis of the data for our collaborators. Although the group has been contacted about characterization of other types of archaeological samples (e.g., obsidian), until now no work has been conducted, with the exception of some preliminary studies of obsidian.

CERAMIC COMPOSITIONAL ANALYSES AT THE NAT GROUP

The first application of NAA to archaeological matrices performed at the laboratory was a study of Peruvian pottery during the early 1980s, by R. Plá in collaboration with the Peruvian Institute of Nuclear Energy (IPEN). The samples consisted of potsherds from different cultures with the objective of seeking possible correlations among them. Shortly afterwards, a study began on archaeological settlements of La Ciénaga and Tafí (Tucumán, Argentina), with archaeologists from several national universities. Potsherds and clay samples were analysed to test a previously formulated hypothesis that La Ciénaga pottery was locally manufactured with clay from the homonymous valley and that similar raw materials were used to manufacture both La Ciénaga and Tafí style pottery. Twelve elements were determined, and for the first time, statistical techniques were used for data evaluation (Cremonte *et al*. 1991). In 1991, sherds and raw materials from the Buenos Aires Province (Central Coast region and other adjacent areas) were analysed. Seventeen elements were determined and multivariate methods were used to evaluate similarities among the samples, previously grouped by classical methods (Aldazábal *et al*. 1993–4). Between 1996 and 1999, under the auspices of the International Atomic Energy Agency (IAEA) Research Program for Nuclear Analytical Techniques in Archaeological Investigations, the NAT Group collaborated with Centro de Estudios Aplicados al Desarrollo Nuclear (CEADEN) of Cuba, analysing 141 samples of aboriginal pottery from the central region of Cuba, which corresponded to sites dated between 1100 and 500 BP (Padilla Álvarez and Celaya González 2003; Padilla Álvarez *et al*. 2003). Also within the framework of the above-mentioned programme, the group worked on the provenance study of ceramic raw materials and artefacts from Chaschuil (Catamarca), in northwestern Argentina to better understand pre-Hispanic social relationships between people living in the cold and arid Puna (3500–4200 m asl) and the adjacent valleys (1500 m asl) (Plá and Ratto 2003). This study was also related with two projects of the University of Catamarca: Chaschuil Archaeological Project and Batungasta Archaeological Rescue Project. This was the beginning of the collaboration with Dr Ratto (University of Catamarca, University of Buenos Aires) on the characterization of ceramic material from Catamarca, on which the laboratory is currently working.

ANALYTICAL PROCEDURES

Sample preparation

When possible, a sherd fragment of about 2 cm^2 (2–3 g) is used. The entire surface is cleaned using a tungsten carbide rotary file. The fragment is then ground in an agate mortar, and the fine powder obtained from grinding is dried in an oven at 105°C for 24 hours. Approximately 100–150 mg of this material is then sealed in a pre-cleaned quartz ampoule for irradiation. Clay samples are ground in an agate mortar and dried in an oven as described for the sherds.

NAA procedure

Samples and standards are irradiated at the RA-3 reactor (thermal flux 6×10^{13} cm^{-2} s^{-1}, 8.5 MW) for 3 hours. Two counts are performed after approximately 7 and 30-day decays, respectively, to quantify the following elements: As, Ba, Ce, Co, Cr, Cs, Eu, Fe, Hf, La, Lu, Nd, Rb, Sb, Sc, Sm, Ta, Tb, Th, U and Yb. The measurements are done with HP Ge detectors (resolution 1.8 keV for the 1332.4 keV Co-60 peak) coupled to an Ortec 919 E Ethernim module, using Gamma Vision software. A software program developed at the laboratory is used to calculate abundances. The National Institute for Standards and Technology's (NIST) standard reference material Coal Fly Ash (SRM 1633b) is used as a calibration standard and NIST's SRM 2709 San Joaquin Soil, China National Research Centre for CRM's GBW07405 (GSS-5 soil) and inter-laboratory standard 'Andesite' are used for quality control.

The reproducibility of the method has been tested by analysing standard reference materials (NIST SRMs 2709 San Joaquin Soil and 699 Brick Clay and USGS AGV). The results from the analysis of the SRMs San Joaquin Soil and Brick Clay were compared with those from the NAA laboratories of Brazil (Instituto de Pesquisas Energéticas e Nucleares—IPEN) and Chile (Comisión Chilena de Energía Nuclear—CChEN). These data are in agreement at the 95% confidence level for Ce, Co, Cr, Cs, Eu, Fe, Hf, La, Sc, Sm, Th and Yb (Munita *et al*. 2001).

Data evaluation

Multivariate analyses are performed on the resulting NAA data to identify significant associations that help to establish the provenance of artefacts and/or other relevant relationships. Data evaluation, carried out on log-transformed data, usually includes correlation, cluster, principal component and discriminant analyses. Prior to the application of any of these procedures, the data are subjected to validation to detect potential analytical problems or errors in data transcription or presentation. For multivariate analyses, only those elements with measurement uncertainties less than 10% and without missing values (or valued below detection limits) are retained, in order to improve the quality of the data matrix. As raw material samples are analysed without previous preparation of bricks, elemental concentration values are increased by 10% for their comparison with sherd analysis results.

PROJECTS RELATED TO ARCHAEOLOGICAL APPLICATIONS

Since the first archaeological application of NAA performed by the NAT Group, researchers from different universities have requested the study of ceramic and raw material samples. Until recently, the laboratory has only received requests for pottery analyses, but we are

Table 1 *Researchers who have worked with the NAT Group in archaeological applications of NAA*

Researcher(s)	*Affiliation*	*Samples*	
		Number	*Site*
M. B. Cremonte and N. Flegenheimer	Universidad Nacional de Tucumán/ Universidad Nacional de la Plata	26	Tucumán, Argentina
V. Aldazábal	CONICET, Argentina	55	Buenos Aires, Argentina
R. Padilla	CEADEN, Cuba	141	Cuba
N. Ratto	Universidad Nacional de Catamarca and Universidad de Buenos Aires	>950	Catamarca, Argentina

prepared to extend our research to the characterization of other matrices. Table 1 provides a summary of investigators with whom the laboratory has collaborated, their affiliation and the numbers of samples analysed. The current absence of an irradiation pneumatic facility limits the amount of work that the laboratory can perform, and it must be remembered that archaeological applications are not the primary line of research for the group.

With the exception of the first analysis of Peruvian pottery, all archaeological studies have been presented at national or international scientific meetings and published. References to this research are included in the references cited section of this paper. The group is currently working on the creation of a database that will include the results from the analysis of Catamarca pottery and raw materials. The laboratory has also taken part in many seminars aimed at educating archaeologists, anthropologists, historians and students on the applications of nuclear analytical techniques to archaeology, art and cultural heritage.

EVALUATION OF THE PRE-HISPANIC INTEGRATION BETWEEN PUNA AND FIAMBALÁ AREAS IN NORTHWESTERN ARGENTINA

The Chaschuil Archaeological Project, a regional research project of the University of Catamarca, in Argentina, had among its objectives the explanation of land use patterns focusing on the study of both environmental and artefactual variability. Under its framework and within the IAEA project already mentioned, a study of ceramic production and distribution by pre-Hispanic societies in Catamarca was initiated in 1996 (Plá and Ratto 2003).

Based on the regional archaeological record structure (Ratto 2000, 2003) the hypothesis was that the Fiambalá Valley and Chaschuil Puna (both in Catamarca) were connected by pre-Hispanic trade routes that facilitated the promotion, retention and reinforcement of social, economic, political and ideological relationships that characterized the regional cultural development from the initial agro-pastoral stages until the Inka period. Thus it is expected that sherds recovered in the Puna area were manufactured using raw materials from the valley rather than with local materials. According to radiocarbon and thermoluminescence dating of archaeological contexts and ceramic artefacts, covering a temporal span of 700 years, both agro-pastoral and state societies transported vessels to the Chaschuil Puna area.

The study area is located at the headwaters of the Chaschuil river and its tributaries and sampling included the upper Chaschuil basin (3500–4200 m asl) and Bolsón de Fiambalá (1500 m asl), where the Batungasta archaeological site is located (Fig. 1). Ceramic material

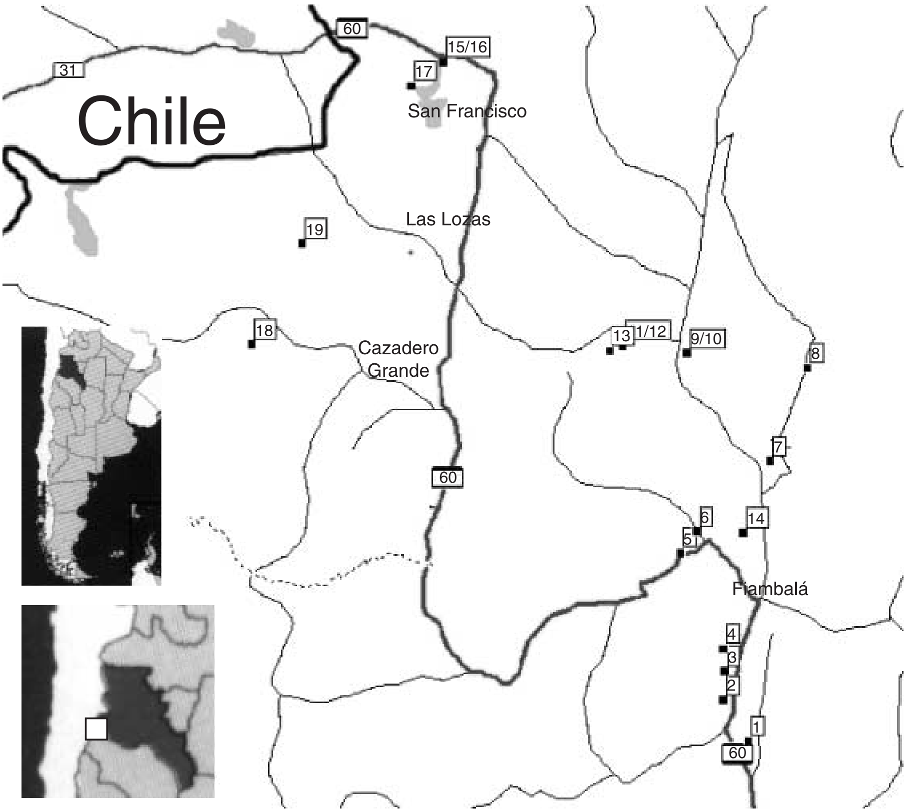

Figure 1 *Sampled archaeological sites in Puna and the valley, Catamarca, Argentina.*

No.	*Archaeological site*	*Function*	*Cultural period*
	Valley		
1	V50-1344 (1365 m asl)	Residential	Formative
2	El Puesto – La Troya (1400 m asl)	Systematic sampling of ceramics	From Formative to Inka
3	Batungasta (1450–1440 m asl)	Residential	Inka
4	Batungasta kilns (1470 m asl)	Productive	Pre-Inka/Inka
5	Huanchín (1700 m asl)	Tombs	From Formative to Inka
6	Guanchincito petroglyphs (1700 m asl)	Ritual	From Formative to Pre-Inka
7	El Horno – Istataco (1730 m asl)	Tombs	From Formative to Inka
8	Tatón 1 (1870 m asl)	Residential	Formative
9	Palo Blanco – NH3 (1930 m asl)	Residential	Formative
10	Finca Justo Pereyra (1900 m asl)	Tombs	Pre-Inka
11	Ranchillos 1 (2340 m asl)	Residential	Inka
12	Ranchillos 2 (2300 m asl)	Residential	Formative
13	Ojo de Agua 1 (2450 m asl)	Residential	Formative
14	Mishma (1700 m asl)	Residential	Pre-Inka/Inka
	Puna		
15	San Francisco (4000 m asl)	Residential	Inka
16	Las Grutas (4000 m asl)	Residential	Formative
17	Las Coladas (4200 m asl)	Residential	Inka
18	Tambería (4000 m asl)	Residential	Inka
19	Ojo de Las Lozas (4000 m asl)	Residential	From Formative to Inka

Table 2 *Distribution of ceramic samples according to area and chrono-cultural period*

Area	*Period*			
	Regional Formative, c. 600–900 BC	*Pre-inka, c. 900–1480* BC	*Inka, c. 1480–1536* BC	*Total sherds*
Chaschuil Puna	47	1	60	108
Fiambalá valley	100	81	16	197
Total sherds	147	82	76	305

from the valley and Puna archaeological sites was analysed, together with clay samples from sites considered as potential raw material provision sources for ceramic manufacture (Ratto *et al*. 2002, 2004, 2006; Plá and Ratto 2003).

A sample of 305 sherds from Puna and valley sites was analysed by INAA. The sherds were classified according to their technological, morphological and stylistic characteristics into different chrono-cultural periods (Fig. 1 and Table 2). Sixty-two clay samples, from Puna and the valleys, also were analysed. Puna and valley clays showed important differences regarding availability and quality for pottery manufacture (Ratto *et al*. 2002, 2004; Plá and Ratto 2003). La Troya river deposits (near the Batungasta site) and others in the vicinity showed excellent properties regarding clay plasticity and workability.

The samples were prepared and analysed as described above. Twenty-two elements were determined, although only compositional data for 16 elements were considered for multivariate statistics. Principal component analysis (PCA) was performed on log-transformed data. After identification and removal of obvious outliers, a second PCA was performed on the dataset. Hierarchical cluster analysis and discriminant analysis were selected for group definition using as input variables the PCA component scores. Mahalanobis distance was used for evaluating the relative probabilities of membership of each sample to the conformed groups. Figure 2 is a bivariate plot based on the first two components for groups identified by discriminant analysis:

(i) group A—mostly pre-Inka/Inka sherds from the valley, with no relation to the sampled clay deposits;

(ii) group B—Puna and valley sherds from different chrono-cultural periods, La Troya clays (specially those from the river lower course) and other clays from deposits at the same altitude;

(iii) group C—mostly clay samples, with only three from La Troya's upper course; and

(iv) group D—three samples from the La Troya area.

The grouping of pottery from Puna and valley areas together with valley clays (especially those from La Troya) points to La Troya as a common production *locus*. In addition to raw material deposits, La Troya presents other type of evidence that reinforces its categorization as a pottery production space: (i) the record of 42 kilns for pottery production (Caletti 2005); (ii) adequate environmental conditions for this activity and (iii) algarroba to serve as fuel (Ratto *et al*. 2002, 2004; Palacios and Brizuela 2005).

The lack of correlation between the regional sample chemical profile and the decorative styles for the different local culture-historic periods also has been observed by Williams and Ratto (2005) at the macro-regional level, for samples considered pre-Inka and Inka due to their techno-decorative characteristics. Moreover, compositionally defined groups include different

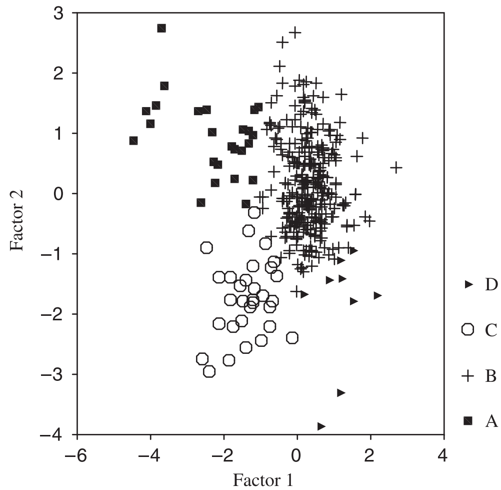

Figure 2 *Representation of the two first components of the PCA of sherds and clays and compositional groups found by discriminant analysis.*

Samples	*Zone*	*Compositionally defined groups*				
		A	*B*	*C*	*D*	*Total*
La Troya clays	Valley	0	14	3	3	20
Other deposits of clay	Valley	0	7	26	0	33
Formative sherds	Puna	1	38	1	2	42
	Valley	1	96	0	3	100
Pre-Inka and Inka sherds	Puna	0	62	0	1	63
	Valley	23	72	1	0	96
Total (sherds and clays)		25	289	31	9	354

technological groups and morphological styles (Ratto *et al*. 2006). This points to raw material sources being the same ones along time, despite other aspects of pottery manufacture that show technical variations (e.g., type of firing, the types of non-plastic inclusions and their size, and surface treatment and decoration of the pottery).

The analytical results and the archaeological evidence support the hypothesis that Batungasta, located in the area of La Troya, served as the ceramic production and distribution centre for Chaschuil Puna and other areas in the valley, during the Inka occupation. According to Ratto *et al*. (2002), it was necessary to have an organized ceramic production system to supply pottery to Puna sites. The interest of the state in maintaining high altitude sites was related to the importance of the domination process, the sanctuaries in the high summits, as well as with obtaining *vicuña* wool (Ratto and Orgaz 2002–4; Ratto 2003). Nevertheless, the exploitation of La Troya area as a source of ceramic raw material can be traced back before Inka occupation, having productive and residential functions for some 700 years. Within this period, regional interaction scales changed but it can be said that the Chaschuil region served as a corridor for the circulation of energy, goods and information for state and formative societies, being one of the multiple circulation routes that integrated the eastern and western territories on both sides of the Andes.

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