

**COMPOSITIONAL STUDIES OF CARIBBEAN CERAMICS:  
AN INTRODUCTION TO INSTRUMENTAL NEUTRON ACTIVATION ANALYSIS**

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*The University of Missouri Research Reactor Center (MURR) initiated a research program in 2003 centered on the chemical characterizations of ceramics from the Caribbean region. This article describes the technique and procedures involved in instrumental neutron activation analysis (INAA), applied to all but one of the studies in this issue. These papers were presented at the 2006 Society for American Archaeology (SAA) 71<sup>st</sup> annual meetings in San Juan, Puerto Rico. The collaborative studies presented are preliminary in nature, but hold promise for addressing a myriad of issues regarding Caribbean prehistory, ranging from island settlement to culture change.*

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The Caribbean region has been one of the most underrepresented areas of the world for chemical characterization studies of ancient material culture. In recent years, compositional studies have begun to address this deficiency (e.g., Bohus et al. 2005; Carini 1991; Crane 1993; Fitzpatrick 2000; Hofman et al. 2005; Lundberg et al. 2002; Padilla et

al. 2003; Padilla et al. 2006; Winter and Gilstrap 1991). This paper introduces one such program initiated at the University of Missouri Research Reactor Facility (MURR) where ceramics from eight projects were investigated to begin building a compositional database of Caribbean ceramics. A description of the techniques and

procedures of INAA applied to the ceramics and clays of most of the papers, is presented followed by an introduction of the different papers and a discussion of the potential of the techniques for addressing issues of Caribbean archaeology. Isendoorn et al.'s paper introduces the archaeometric program at Leiden University where various archaeometric techniques have been used for the study of Caribbean ceramics (see also Hofman et al. 2005).

The senior author initiated this archaeometric project on Caribbean ceramics in 2003 while a postdoctoral researcher at the Research Reactor Center. Several leading Caribbean researchers based at US academic institutions were invited to participate in the study and were offered 50 free analyses in exchange for participating in the SAA conference session. The long-term goal of the study is to address some of the major anthropological questions of the region by building an elemental database of the most common artifact type found in the historic and prehistoric Caribbean archaeological contexts—ceramics. The technique has been applied successfully to numerous other cultural regions in the Americas, notably the American Southwest and Mesoamerica (Glowacki and Neff 2002; Nichols et al. 2002). INAA has allowed for the exploration of the changing roles of markets and the development of preindustrial markets in the Basin of Mexico. In the American Southwest, INAA has become a useful tool in understanding exchange, migration, social identity, and economic organization.

Financial support for the INAA studies presented in this issue was provided by the National Science Foundation (BCS-0102325) and the US Department of Energy Office of Nuclear Energy, Science and Technology

(Award No. DE-FG07-03ID14531) to the Midwest Nuclear Science and Engineering Consortium under the Innovations in Nuclear Infrastructure and Education program. Isendoorn et al.'s X-ray fluorescence (XRF) analyses and fieldwork were funded by the NWO VIDI-project "Mobility and exchange: dynamics of social, material and ideological relations in the pre-Columbian insular Caribbean" directed by Professor C. L. Hofman and by the Byvanck Fonds.

The Yale University Peabody Museum of Natural History and the late Professor Irving Rouse graciously facilitated Christophe Descantes' compositional studies of Indian Creek ceramics from the West Indian island of Antigua. The INAA study of these data was submitted for publication in the Proceedings of the 21st Congress of the International Association for Caribbean Archaeology (IACA) in Trinidad and Tobago (Descantes et al. 2007). Antiguan data presented in the paper above are used in this introductory paper to illustrate the INAA technique and the reduction of the data.

In addition to the compositional programs at Leiden University and MURR described in this volume, there have been other recent compositional studies of Caribbean ceramics—most notably the work of Román Padilla et al. at the Centro de Aplicaciones Tecnológicas y Desarrollo Nuclear (CEADEN) in Cuba, and of Magdalena Mackowiak de Antczak and Andrzej Antczak at Simón Bolívar University in Caracas, Venezuela.

The INAA work of Padilla et al. (2003) from CEADEN successfully combined INAA and scanning electron microscopy X-ray analysis (SEM–EDX) to study Cuban pottery from prehistoric and historic assemblages. Incorporating petrographic analyses in their

work, they confirmed that different clay regions in central Cuba have different clay compositions. When investigating the provenance of Majolica ware recovered at Cuban sites, Padilla et al. (2003) argue that the pottery originates from Spain, Old Havana in Cuba, and possibly Veracruz, Mexico. Recently, Padilla et al. (2006) combined two energy dispersive X-ray fluorescence methods—improved sensitivity polarized X-ray fluorescence analysis (EDPXRf) and radioisotope X-ray fluorescence (R-XRF)—for fast and non-destructive analyses of ceramic sherds. They found the chemical measurements were comparable to that of INAA, provided the surface of the sherd was flat and had no evident signs of physical alteration, such as cracks or cavities.

Similarly, Magdalena Mackowiak de Antczak and Andrzej Antczak from Simón Bolívar University in Caracas, Venezuela, collaborated with the Institute of Isotope and Surface Chemistry in Budapest to determine specific areas of production of Los Roques Archipelago ceramic figurines. As in other archaeometric studies into Caribbean material culture, various analytical techniques, such as INAA, total reflection X-ray fluorescence (TXRF), and prompt gamma activation analysis (PGAA) are combined with traditional petrographic analyses to not only provenance the stylistically diverse ceramic figurines, but also to investigate the social and ideological aspects of the figurines in their respective archaeological deposits. Preliminary results reported indicate that similar styled figurines from the Los Roques Archipelago and from the Lake Valencia region in Venezuela differ significantly in chemical composition (Bohus et al. 2005:255).

### ***The INAA Technique***

Instrumental neutron activation analysis is a powerful quantitative analytical technique that has been widely applied in archaeological studies for the last 50 years (Glascock and Neff 2003; Neff 2000; see papers in Speakman and Glascock 2007). The precision and accuracy of this radiometric technique in generating empirical data for major, minor, and trace elements of objects of interest makes it one of the most reliable compositional characterization techniques, especially useful for addressing questions of provenance or origins.

In brief, INAA involves activating the nuclei of objects by bombarding them with neutrons generated from a nuclear reactor (Figure 1). Once activated, nuclei emit gamma rays with characteristic energies that can be measured and attributed to specific isotopes. Quantitative INAA data can be generated by including known reference standards in the analyses (see Glascock 1992).

### ***Sample Preparation***

Standard MURR procedures were used in the INAA of the Caribbean ceramics and clay samples (see Glascock 1992). Fragments of about 1cm<sup>2</sup> were removed from each sample and abraded using a silicon carbide burr in order to remove glaze, slip, paint, and adhering soil, thereby reducing the risk of measuring contamination. Specimens were then washed with deionized water before being crushed into fine powder in an agate mortar. Where possible a portion of each specimen was retained, unpowdered, for the MURR archive of analyzed ceramic fabrics. Powdered samples were oven-dried at 100 degrees Celsius for 24 hours. Portions of approximately 150 mg were weighed and

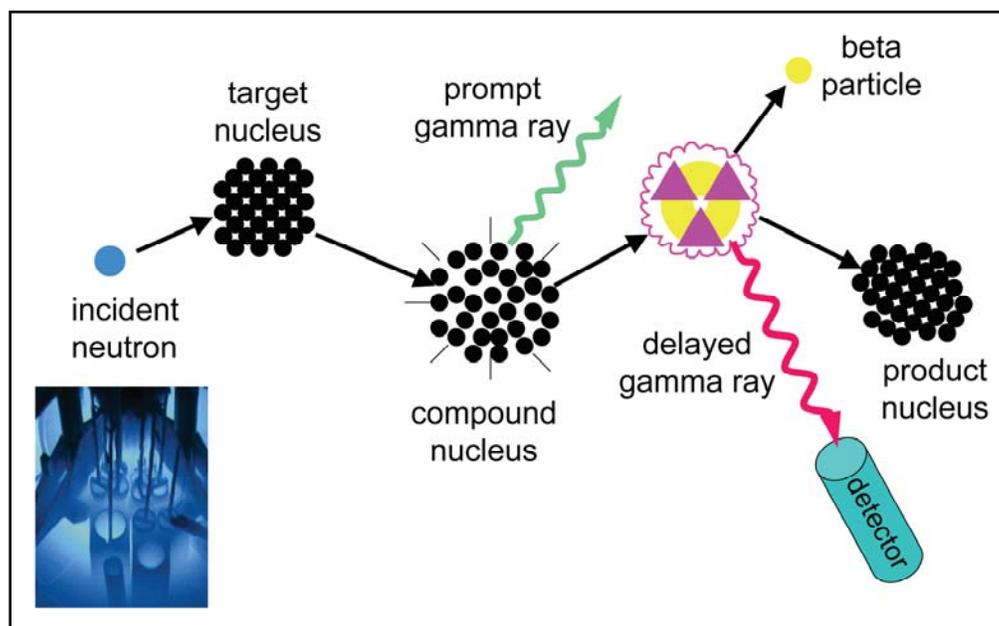


Figure 1. Schematic diagram of instrumental neutron activation analysis (adapted from Glascock 1998:100).

placed in small polyvials used for short irradiations. At the same time, 200 mg of each sample were weighed into high-purity quartz vials used for long irradiations. Along with the unknown samples, reference standards of SRM-1633a (coal fly ash) and SRM-688 (basalt rock) were similarly prepared, as were quality control samples (e.g., standards treated as unknowns) of SRM-278 (obsidian rock) and Ohio Red Clay (an in-house standard).

#### ***Irradiation and Gamma-Ray Spectroscopy***

INAA of ceramics at MURR consists of two irradiations and three gamma counts. As discussed in detail by Glascock (1992), a short irradiation is carried out through the pneumatic tube irradiation system (Figure 2). Samples in the polyvials are sequentially irradiated, two at a time, for five seconds at a neutron flux of  $8 \times 10^{13} \text{ n cm}^{-2} \text{ s}^{-1}$ . The 720-second count yields gamma spectra containing peaks for nine short-lived

elements: aluminum (Al), barium (Ba), calcium (Ca), dysprosium (Dy), potassium (K), manganese (Mn), sodium (Na), titanium (Ti), and vanadium (V). The samples encapsulated in quartz vials are subjected to a 24-hour irradiation at a neutron flux of  $5 \times 10^{13} \text{ cm}^{-2} \text{ s}^{-1}$ . This long irradiation is analogous to the single irradiation utilized at most other laboratories. After the long irradiation, samples decay for seven days, and then are counted for 1800 seconds (the "middle count") on a high-resolution germanium detector coupled to an automatic sample changer. The middle count yields determinations of seven medium half-life elements, namely: arsenic (As), lanthanum (La), lutetium (Lu), neodymium (Nd), samarium (Sm), uranium (U), and ytterbium (Yb). After an additional three- or four-week decay, a final count of 9,000 seconds is carried out on each sample. The latter measurement yields the following 17 long half-life elements: cerium (Ce), cobalt (Co),

chromium (Cr), cesium (Cs), europium (Eu), iron (Fe), hafnium (Hf), nickel (Ni), rubidium (Rb), antimony (Sb), scandium (Sc), strontium (Sr), tantalum (Ta), terbium (Tb), thorium (Th), zinc (Zn), and zirconium (Zr). The element concentration data from the three measurements are tabulated in parts per million using Microsoft Excel.

**Quantitative Analysis of the Chemical Data**

The following section is a brief description of the data reduction and analytical procedures used in the chemical compositional analysis. See Neff (1994, 2000) for more detailed information. As is customary in ceramic provenance studies at MURR (Bishop and Neff 1989), the data are converted to base-10 logarithms of concentrations. Use of log concentrations rather than raw data compensates for differences in magnitude between major elements. Chemical data values for the 33

elements in the analyzed samples are examined prior to identifying compositional groups. Specimens with anomalous concentrations are treated as outliers and rejected from subsequent statistical procedures. In addition, elemental abundances that are non-existent or below detection limits in many of the samples are also dropped from further analyses. The elimination of nickel measurements is not uncommon in the chemical analysis of ceramics in general and in Caribbean ceramics in particular. The last procedure involves searching for a pattern of calcium concentrations greater than 1% or 10,000 parts per million. The high calcium abundances are most likely a result of either of the original clay sources or a calcium-rich temper. A correction factor is applied to all of the elemental data to counter the dilution effect of calcium on the other elemental abundances (see Cogswell et al. 1998). As in

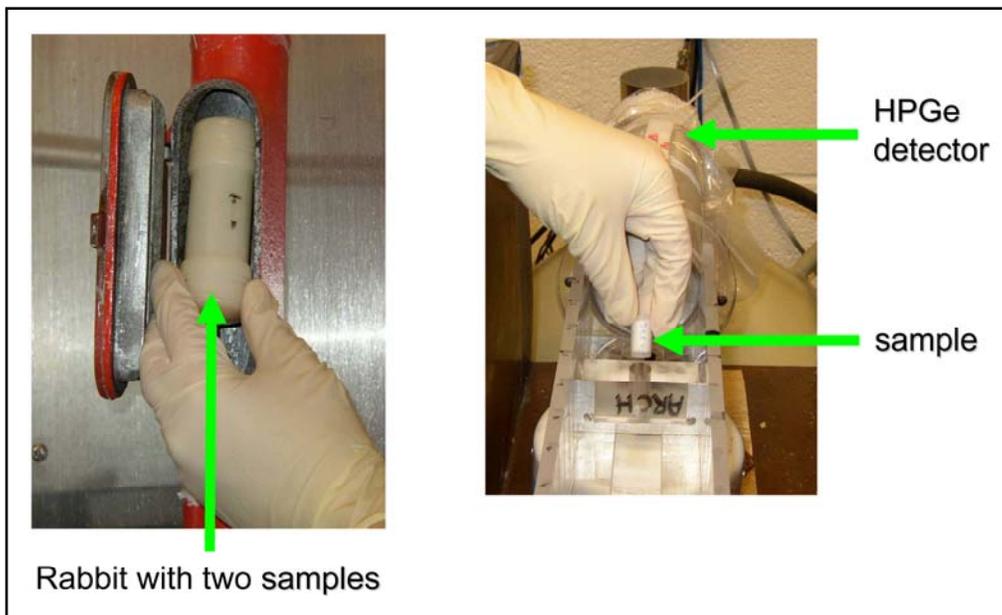


Figure 2. Short irradiation of a rabbit carrying two samples in a pneumatic tube system (left) and the measurement of gamma-ray emission in front of a high-resolution germanium detector (right).

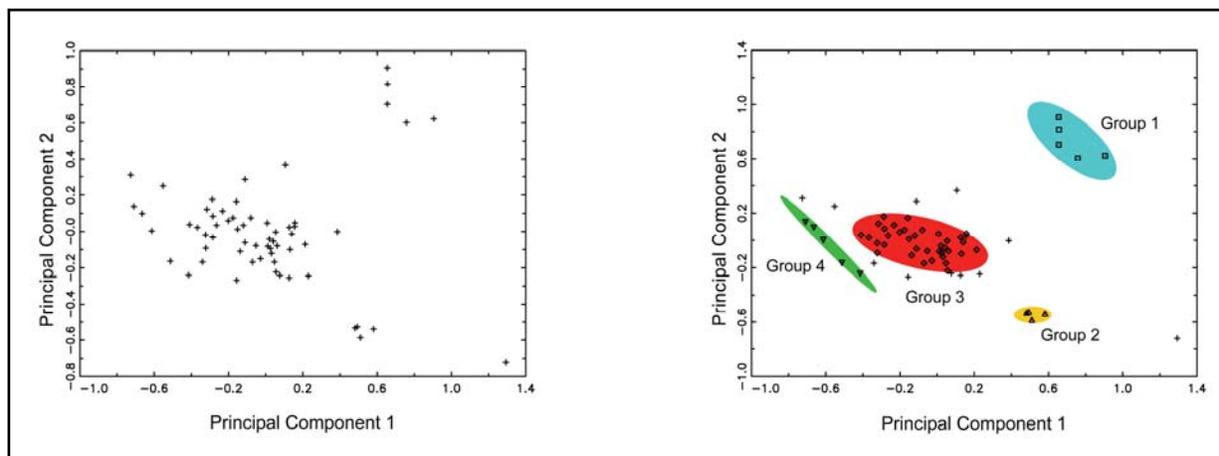


Figure 3. Plot of principal components 1 and 2 displaying the ceramic INAA data from the Indian Creek Site, Antigua (left) and plot of the same data with identified compositional groups (right). Unassigned specimens (+) are not labeled.

previous treatments of ceramics with enriched calcium concentrations, we eliminated the elemental concentrations of calcium and strontium. A calcium correction factor was applied to the ceramic data from the Dominican Republic and Puerto Rico.

The goal of quantitative analysis of the chemical data is to recognize compositionally homogeneous groups (that are hopefully anthropologically meaningful) within the analytical dataset. Based on Weigand *et al.*'s (1977) "provenance postulate", such groups are assumed to represent geographically restricted sources or source zones. The location of sources or source zones may be inferred by comparing the unknown groups to knowns (source raw materials) or by indirect means. Such indirect means include the "criterion of abundance" (Bishop *et al.* 1982) or arguments based on geological and sedimentological characteristics (e.g., Steponaitis *et al.* 1996).

Initial hypotheses about source-related subgroups in the compositional data are derived from the application of pattern-recognition techniques to the chemical data.

Principal components analysis (PCA) is used to recognize patterns, that is subgroups in the compositional data (Figure 3). PCA provides new reference axes that are arranged in decreasing order of variance subsused. PCA can be used in a pure pattern-recognition mode, i.e., to search for subgroups in an undifferentiated data set or in a more evaluative mode to assess the coherence of hypothetical groups suggested by other criteria, such as archaeological context and decoration.

Biplots are used to display both the variables (elements) and objects (individual analyzed samples) on the same set of principal component reference axes (Figure 4). Displaying objects and variables on the same plots makes it possible to observe the contributions of specific elements to group separation and to the distinctive shapes of the various groups.

The Mahalanobis distance statistic is used to statistically test the separation between groups or between individual points and groups on multiple dimensions (see Bieber *et al.* 1976; Bishop and Neff 1989; Harbottle

1976; Neff 2001; Sayre 1975. Mahalanobis distance takes into account variances and covariances in the multivariate group and is analogous to expressing distance from a univariate mean in standard deviation units. Similar to standard deviation units, Mahalanobis distances can be converted into probabilities of group membership for each individual specimen (e.g., Bieber et al. 1976; Harbottle 1976).

Mahalanobis distance-based probabilities of group membership for members in small groups may fluctuate dramatically depending on whether or not each specimen is assumed to be a member of the group to which it is being compared. This limitation can be circumvented by cross-validation or "jackknifing", that is, by removing each specimen from its presumed group before calculating its own probability of membership (Baxter 1992; Leese and Main 1994). All probabilities discussed in each of the papers in this issue were cross-validated.

Mahalanobis distances can be calculated for the log concentrations and the principal components extracted from the variance-covariance or correlation matrix of the complete data set. Calculating Mahalanobis distances on the principal components permits us to reduce the dimensionality of the data set and statistically test the separation of the chemical groups, which is crucial when dealing with small groups. We use enough principal components to subsume approximately 90% of total variance in the data set. Typically, elemental plots are provided in the analysis to demonstrate that the identified compositional groups are not an artifact of the PCA algorithm (see Figures 4 and 5).

### ***Potential of INAA studies***

The compositional study of ceramic materials from archaeological sites promises to shed light on some of the major archaeological questions of the region. Such questions include ancient population movements within the region, the development of exchange networks both within and among various island archipelagoes, and local adaptations and historical developments from ca. 4000 BC up to and including the time of Euroamerican contact.

Material culture in the Caribbean has long served as a marker for identifying cultural traditions and modeling population movements (Keegan 1995; Rouse 1992). In regards to the original movements of preceramic peoples into the Antillean region, many areas have been suggested as possible sources (e.g., Cruxent and Rouse 1969; Veloz and Vega 1982; Wilson et al. 1998). The Yucatan Peninsula is favored as the most likely source for the early people who settled the Greater Antilles (Keegan 2000). In contrast, the later horticultural ceramic-producing peoples who populated the Lesser Antilles sometime during the first millennium BC are hypothesized to be migrants from northern South America (near the mouth of the Orinoco River). Compositional analyses of the material culture, and where possible, isotope ratios (e.g., Sr) of human bone and teeth, will contribute to our understanding of the complex migration movements into the region, and serve as an important counterpoint to previous migration hypotheses based on artifact attributes and historical linguistic studies. Compositional data for ceramics from early assemblages can provide quantifiable evidence and track technological changes during the rapid island

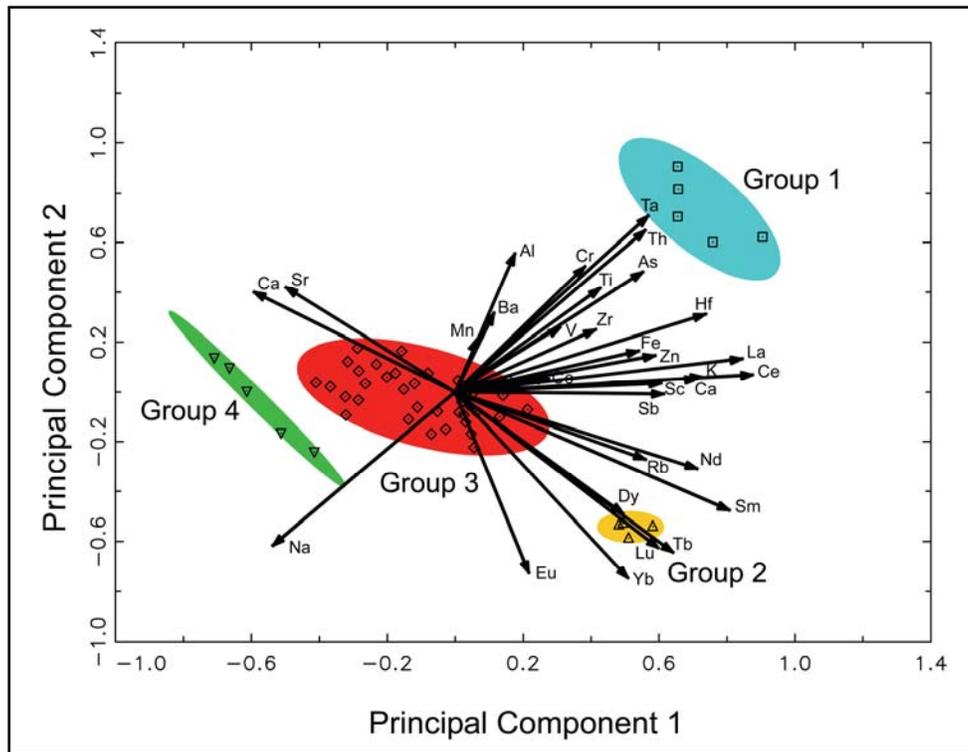


Figure 4. Correlation matrix PCA biplot of principal components 1 and 2 showing the four compositional groups identified in the Indian Creek Site ceramic sample. Ellipses represent 90% confidence level for membership in the groups. Unassigned specimens are not shown.

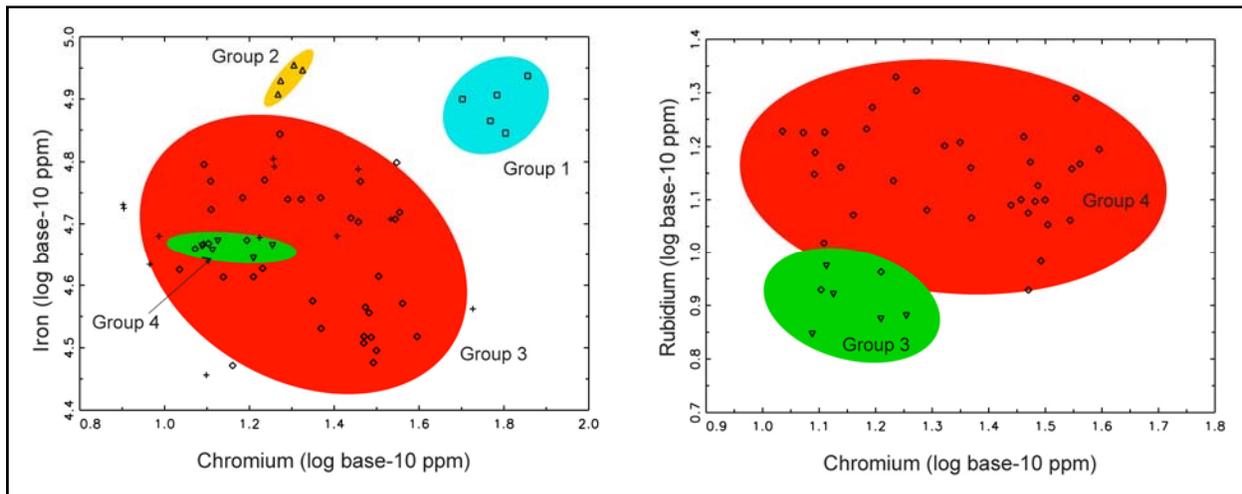


Figure 5. Left: Plot of base-10 logged chromium and iron concentrations of the four ceramic compositional groups at the Indian Creek Site, Antigua. Ellipses represent 90% confidence level for membership in the groups. Unclassified samples (+) are not labeled. Right: bivariate plot of base-10 logged chromium and rubidium concentrations displaying the chemical distinctiveness of compositional Groups 3 and 4. Ellipses represent 90% confidence level for membership in the groups. Unclassified samples are not shown.

settlement of the Caribbean. In particular, the acquisition of ceramic and clay compositional data can allow one to identify the possible interactions between people of new island settlements with the people from whence they came. Finally, studies using INAA can also contribute insights into the origin of ceramic technological behaviors of African slave societies in the Caribbean by investigating comparable technologies in West Africa.

Constructing past exchange models is a major task facing Caribbean archaeologists (e.g., see Crock and Petersen 2004; Hauser 2001; Knippenberg 2006). Exchange networks are extensive in Caribbean prehistory; ceramics, cherts, and other materials found in the archaeological record are important sources of evidence for identifying past exchange behaviors. Petrographic analyses of ceramics in the region have long provided invaluable evidence for addressing questions of ceramic production and exchange, and still have a vital role to play as a complimentary technique to finer-grained analytical techniques. Detailed studies that employ INAA and petrographic analysis to study the material culture of the peoples of the Caribbean contribute to our understanding of exchange networks, which are important for developing and testing hypotheses about the origins, the operation, and the transformation of exchange systems.

Permeating all aspects of island life, exchange initiates interaction between people and is an important dynamic in discussions of culture change. Exchange studies based on compositional data can provide insights on cultural processes of ethnogenesis, enculturation, colonialism, and resistance in the prehistoric and recent past. Leiden University's Archaeology Program presently

heads several Caribbean research projects that employ state-of-the-art archaeometric techniques, such as thermal ionization mass spectrometry (TIMS), to gain insights into the movement of objects and their impacts on the societies involved.

Compositional analyses of ceramics can provide insights into the technologies of past peoples (e.g., Curet 1997). Compositional analyses of undecorated and decorated wares aimed at understanding behavioral processes, from procurement of clay at specific geologic sources to the deposition of broken ceramic vessels in the archaeological record can allow archaeologists to explain local adaptations and historical developments in the societies of the region. INAA analyses of Caribbean ceramics permit an investigation into the technological attributes of the decoration-based typological categories of the ceramics. A diachronic perspective based on the composition of particular ceramic wares allows us to investigate issues of the so-called decrease in ceramic quality as well as the great cultural diversity in the West Indies around AD 800 (e.g., see Keegan 2000:155). We know that dramatic changes in subsistence and pottery decoration mark the boundary between Saladoid and Ostionoid cultures of Puerto Rico (Keegan 2000:152). Is this shift represented in the compositional record of the sherds? It has been argued that cultural pluralism is a key factor in understanding the emergence of Taíno chiefdoms (Wilson 1999:2). Is this pluralism represented in their ceramic recipes? In conjunction with the ethnohistoric sources (see e.g., Hofman and Bright 2004) that exist for the region, the compositional studies we propose stand to gain from understanding pottery manufacturing in changing and social and cultural environments and understanding how pottery was negotiated in African, European, and indigenous societies.

Relying on small data sets, the papers in this issue use compositional data from ceramics to address some of the major questions of the region and offer preliminary interpretations. All of the studies presented here are works in progress, and will undoubtedly be expanded upon by enlarging the sample sizes of the clays and ceramics, and applying various analytical techniques to the research questions. The work by Isendoorn et al. presents preliminary results of a case study on St. Lucia to address wider concerns of reconstructing contact and/or distribution networks in the Lesser Antilles during the Ceramic Age. Their micro-regional case study involving three archaeological sites in southern St. Lucia finds that the majority of ceramics originate from local clays in the region. Siegel et al.'s exploratory paper attempts to identify compositional signatures for ceramics from various periods in Puerto Rican prehistory to investigate exchange. They conclude that ceramic production and exchange remained at the local domestic level despite the changing social and political contexts of the island. Crock et al. with a dataset of Late Ceramic Age ceramic sherds from Anguilla in the British West Indies and the Salt River Site in St. Croix of the U.S. Virgin Islands depend on stylistic analyses and INAA compositional work to suggest a degree of specialized production and the use of a limited number of ceramic recipes for both island communities. They conclude that ceramic recipes were limited in both island contexts; the ceramic sample from St. Croix appeared to have more of a local origin than that from Anguilla.

Conrad et al. use the compositional heterogeneity of Taíno ceramics from the Dominican Republic to argue that the site of La Aleta served as a ceremonial center for a regional rather than a local population. Fitzpatrick et al. attempt to reconcile

differences in their petrographic and chemical characterization interpretations of ceramics on the island of Carriacou in the southern Grenadines. To offer preliminary insights into the production and exchange of ceramics on the island of Carriacou in the southern Grenadines from ca. AD 400–1200, they suggest several different possible exotic sources for their ceramic sample.

Several papers in the volume address issues in the more recent historical past. Kelly et al. investigate low-fired earthenwares to address issues of exchange and interaction by focusing on the enslaved peoples of African descent in Guadeloupe during the French Colonial Period of the eighteenth and nineteenth centuries. Preliminary conclusions on the heterogeneity of ceramic recipes imply complex interisland trade of industrial wares. Ahlman et al. examine the wares of enslaved Africans at Brimstone Hill Fortress National Park on St. Kitts from contexts dating from 1790–1850 for insights in their systems of production and exchange. Incorporating possible local clay sources in their compositional analysis, and using a mineralogical and chemical compositional approach, they interpret that most of the ceramics in their sample were locally made. They also suggest the possibility of socio-economic differences amongst the enslaved Africans at Brimstone Hill, where slaves with specialized tasks at the fortress had the means to purchase more expensive non-local pottery than the less skilled laborers, such as plantation slaves. Finally, Hauser et al. focus on the mineralogical and chemical compositions of Yabba ware for insights into the eighteenth century craft production of enslaved and free Jamaicans. At this preliminary stage, it appears that ceramic pot recipes on the north and south coasts and the central part of the island were similar,

indicating a larger than expected scale of production.

### **Conclusion**

Caribbean pottery technology has received relatively little attention (Curet 1997:497). To address this need, several analytical programs have begun to use compositional data of ancient and historic Caribbean material culture to address the salient research questions of the region. MURR has initiated a new program for the chemical characterization of ceramics using INAA, as well as other analytical techniques at its disposal such as inductively coupled plasma mass spectrometry (ICP-MS), energy dispersive X-ray fluorescence spectrometry (EDXRF), and portable X-ray fluorescence spectrometry (PXRF). All of the papers in this volume were presented at the 2006 Society for American Archeology (SAA) annual meetings in San Juan, Puerto Rico, in a session entitled "An Exploratory Study into the Chemical Characterization of Caribbean Ceramics: In Memory of James B. Petersen. The INAA compositional data for eight of the nine papers in this volume were generated at MURR; the XRF data for one paper was generated at Vrije Universiteit Amsterdam.

The research reported in this conference session and the baseline ceramic compositional data generated for the Caribbean will serve as a foundation for future research into the prehistory of this region. We are optimistic that archaeologists are beginning to recognize the value of integrating such studies for understanding important aspects of prehistoric human and social dynamics. Our hope is that we will foster greater collaboration between archaeology and the physical sciences in this region of the world by demonstrating the

intrinsic value of provenance studies to understanding past human social dynamics.

### **Acknowledgments**

We express our gratitude to the contributors in this volume, whose participation has made this collaborative study a success. We thank Ronald Bishop of the Smithsonian Institution who served as discussant for the session at the SAA annual meetings in San Juan, Puerto Rico and for his invaluable comments on the papers in this volume. They provided a wide array of research questions to the archaeological ceramic sherds collected from diverse Caribbean island locations and varied temporal contexts. We also thank Jonathan Dake, Mark Hammond, Nicole Little, Rebecca Schmidt, and Tessa Schut for carrying out the preparation and irradiation of samples in all of the INAA studies. Operating support for the MURR Archaeometry Laboratory was provided by grants from the National Science Foundation. The INAA work was also partially funded by the US Department of Energy Office of Nuclear Energy, Science and Technology Award (No. DE-FG07-03ID14531) to the Midwest Nuclear Science and Engineering Consortium under the Innovations in Nuclear Infrastructure and Education program. Finally, we would like to acknowledge the important support and contributions of the late Professors Irving Rouse and James B. Petersen.

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