ARCHEM Project

▪ Brandeis University ▪ 415 South St ▪ Waltham, MA 02454 ▪ USA ▪ 1.781.736.2689 ▪
▪ INSTAP Study Center for East Crete ▪ Pacheia Ammos ▪ Ierapetra 72200 ▪ Crete, Greece ▪ 30.69868.55354 ▪
▪ akoh@brandeis.edu ▪

Andrew J. Koh
Director

Sampling and Storage Protocol

Introduction to ARCHEM and Organic Residue Analysis

Organic Residue Analysis (ORA) involves three major steps to produce qualitative and quantitative data of archaeological significance: (1) Extraction; (2) Instrumentation; (3) Interpretation.

Extraction isolates a measurable quantity of ancient organic residues (typically in 20mL scintillation vials), which are then accessible for a multitude of analytical techniques in perpetuity. During extraction, fragments of objects are boiled in solvents and returned unharmed, if cleaner. Size and a high degree of preservation sometimes necessitate the "swishing" of more intact objects to extract residues (see below).

Instrumentation involves the separation and characterization of the individual, organic compounds that compose each sample using analytical instruments (usually gas chromatography combined with mass spectrometry, or GC-MS, for ARCHEM).

Finally, one must interpret this data in order to quantify and identify organic compounds, hypothesize which commodities produced them (e.g. wine, olive oil, perfumed oils, food, etc.), and glean what impact this might have on interpreting the surrounding space. Interpretation is the most difficult and time consuming of the major steps and often requires close collaboration with those studying the contexts in which the residues were found (see below). Due to age and decomposition, ancient commodities tend to leave a different signature of surviving compounds than one might find with modern cognates. One must also keep in mind that chemistry will tell us which organic compounds were present when objects were excavated, but it will not directly identify the ancient commodities from which they derived or the timeframe in which different commodities originally resided in objects. This is why an intimate knowledge of the flora, fauna, and cultural practices of the people being studied is vital for accurate and timely interpretations of organic residues.

ARCHEM has collected thousands of samples from as early as the Neolithic Age to recent ethnographic samples with a particular focus on antiquity. The goal is to preserve and maintain a comprehensive library of organic residues for subsequent chromatographic and spectrometric analysis at offsite chemistry facilities guided by specific research plans devised in conjunction with project directors. These analyses currently take place at the GC-MS Lab in the Brandeis University Department of Chemistry. Once data is produced, it is carefully interpreted to determine any significance for the archaeological research questions at hand.

Which Objects are Good Candidates for Organic Residue Extraction?

In order to insure the acquisition of quantitatively and qualitatively significant samples, one must evaluate the level of preservation and potential contamination of each object. One of the primary concerns is that the objects be sampled before any cleaning or conservation with as minimal of handling as possible. The longer one waits before sampling and isolation, the greater the chance for contamination and degradation.

Organic residues can be extracted from both whole (swishing) and fragmentary objects (boiling). Fragments are optimally chosen from the base, rim (especially cookpots), and body – in that specific order. Care must be taken to ensure that samples taken from multiple fragments originate from a single object – i.e. they all join. If unsure, choose only a single fragment from the base at a location where it joins the body (typically the low point where organics pool). For the swishing of largely intact vessel, solvents are heated in a beaker as usual, but they are then poured in small volumes into the vessel and swished around before filtration into separate samples, which are carefully noted in order (the earlier samples are more likely to suffer from contamination).

Past work has revealed difficulties with objects exposed to the elements or irrigation in the distant or recent past, though there is the potential for fat-soluble/non-polar compounds to survive embedded in the fabric matrix. Unless these objects are unique or particularly important, they are best passed up in favor of better-preserved examples. These exposed objects rarely provide a clear picture of their contents and the limited sample quantities they produce often complicates ongoing research and precludes the potential for reproducing and verifying results.

Handling Objects for Sampling

Potential objects should be collected with clean and inert cloth, or gloves made of latex rubber, Teflon, or fabric, and subsequently wrapped in several layers of aluminum foil. Clean trowels can be used but attempts should be made to keep soil between the object and trowel. Soil samples from the same context can help determine if it is a source of contamination, but past studies have shown this to be a rare phenomenon. Largely intact objects should be treated in a similar manner to fragments. If size is an issue for wrapping with foil, other options include new, inert bags that are not derived from plastics (e.g. cloth). The key is to avoid direct human contact and plastic bags (pliable, clear plastic is sure to transfer plasticizers). Recently excavated objects should be allowed to breathe to prevent excessive sweating. Objects should be clearly labeled with contextual and other information relating to how it was found and isolated, with photos in situ an added bonus. Preliminary trench reports are most helpful. Some forms of contamination include exposure to nicotine, suntan lotion, plasticizers, and a number of other common substances found at an excavation site. These forms of contamination can significantly obscure instrumentation data and subsequent interpretation. Objects suspected of coming into contact with any such contamination should be duly noted and avoided for organic residue analysis if at all possible in favor of better examples, but we understand this is not always possible.

What Happens Next?

After residue samples are isolated and documented, a research plan must be crafted in order to expedite instrumentation and interpretation. Due to high costs for instrumentation and the time involved for interpretation, arbitrarily subjecting dozens of samples through these final steps is neither expeditious nor generally recommended. Anywhere from a handful to several dozen samples are a manageable and useful study size for an ORA research plan, which typically targets assemblages or typology, both diachronic and from a single chronological horizon. During this process, references to publications relevant to the study are always welcome.

Due to the close collaboration necessary between ORA and excavators to interpret the meaning of identified compounds, the latter should be in an active stage of study for the objects in question. ORA, unlike disciplines such as faunal analysis and paleobotany that have ancient remains in their fundamentally unaltered state, does poorly when isolated without significant collaborative feedback at various stages. Until a basic study of the objects and their contexts has been accomplished, a simple "yes-no" analysis can be conducted that verifies the existence of organic residues, but interpreting compounds beyond this point bereft of feedback can be unnecessarily time consuming and ineffectual. Feedback can also rule out issues related to contamination, bleeding between sample runs during instrumentation, and other hindrances to interpretation. After this period of collaboration, a preliminary interpretation of the target objects is produced, which can then be further nuanced in the future both before and after final publication.