Physico-Chemical Fingerprinting Techniques: Provenance, People and Landscape

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Landscape archaeology is about the interplay of cultural practices in their social, environmental and ontological settings over time and across multiple spatial and temporal scales, as this Element outlines. The incorporation of scientific analyses into archaeological investigations, particularly when dealing with landscape perspectives, therefore requires the integration of a range of scales of spatial and temporal data.

Physico-chemical fingerprinting techniques are used in sourcing studies to trace peoples' mobility, not only physically (the movement of people and goods, both raw materials and shaped material culture) but also ontologically (the movement of ideas through shared cosmologies such as Australian Aboriginal Songlines or Dreaming tracks actualised in ritual activities shared between groups). Cultural landscapes are, after all, conceptual spaces where '(t)he meaning of … goods and the significance of their source location are explained and maintained over time by the stories that also map the symbolic and geographical world of … long distance transactions' (McBryde 1997b: 605). In this explainer, I focus on the physico-chemical analyses used in archaeological sourcing studies. I discuss some of the impacts that the take up of commercially manufactured portable, handheld instrumentation like portable X-Ray Fluorescence (pXRF) spectrometers has had on the number and quality of archaeological sourcing investigations. Unapologetically, I draw on many examples from archaeological pigment research and the regional records of Australia with which I am most familiar. The overarching principals remain the same, irrespective of the materials characterised.

Physico-chemical approaches—analyses of the geochemical, structural and morphological properties of material culture and the raw materials from which it is made—are used by researchers engaged in sourcing studies to trace human mobility, aiming to link people with geographic points in the landscape. An example is when raw materials were transported by people outside their natural (geological/geomorphological) occurrences, leaving material evidence of that movement in the landscape such as in the form of manuports or modified artefacts (Mills et al. 2022; Scadding et al. 2015). Physico-chemical analyses can also provide insights into past technologies where evidence of processing—the deliberate modification of raw materials—is preserved (MacDonald et al. 2019). Physico-chemical characterisations can demonstrate relationships between raw materials, artefacts, and their transport. In the 'Goldilocks Zone' (i.e. where all necessary conditions are met) of sourcing archaeological materials (also known as 'provenance investigations'), physico-chemical characterisations help quantify the distance and direction that goods and materials have been moved by people, providing insights into human mobility and social interactions, including the socialisation of landscapes (by convention, we use the shorthand 'landscapes' to refer to the full range of 'scapes' including seascapes; see e.g. Sheppard et al. 2013) (Barberena et al. 2019; MacDonald et al. 2018; Sanhueza et al. 2021).

Sourcing studies have taken two main trajectories, concentrating either on archaeological assemblages to describe discrete compositional groups (e.g. Dayet et al. 2014), or on geological features of raw material sources to build datasets against which artefact compositions can be compared (e.g. MacDonald et al. 2013; Velliky et al. 2019). Of course, provenance investigations aim to integrate the characterisation of archaeological materials and 'raw' resources, to trace the movement of resources, material culture and thereby people across landscapes (Barberena et al. 2018; Dayet et al. 2016; MacDonald et al. 2018; Sanhueza et al. 2021). Recognising the different scales at which the physico-chemical signatures of regional geological and archaeological assemblages operate is critical to the success of such endeavours (Linse 1993; MacDonald et al. 2011; Sanhueza et al. 2021).

'Provenance' describes the systematic observation of a raw material or artefact's physico-chemical composition, and characterises that composition's association with the physico-chemical composition of the resource from which it has been manufactured (Pollard et al. 2007). The underlying principle of physico-chemical sourcing is that material culture and/or raw materials can be assigned to the spatially restricted resource from which it was extracted or manufactured, so long as the composition difference *between* source materials exceed the compositional variation *within* a given source material (Weigand et al. 1977; Neff 2002).

The degree of precision and accuracy to which this will be possible will depend on the degree to which a geographically discrete resource is distinguishable from other sources of the same raw material (here I distinguish 'source' from 'quarry', where the former simply refers to the location of a particular type of raw material whereas a quarry is a specific extraction zone of that raw material). The central tenet of sourcing studies is that similarity in the composition of artefacts and raw materials is evidence for their common geographic origin. Therefore, provenance of archaeological artefacts can be assigned to a raw material source or a group of other artefacts without the necessity to identify a geographic find-spot (known as 'provenience') (e.g. Dayet et al. 2016). In other words, sourcing studies can establish relationships between raw materials and artefacts regardless of whether the raw material source is ever found and documented. Indeed, sensitive modern scientific techniques can describe minor compositional constituents, fundamental in recognising the geomorphic contexts of unknown resources, that can be used to identify likely locations within the landscape from which archaeomaterials were extracted, as well as characterising minute components to differentiate between neighbouring resources (Brand et al. 2019; Glascock 2020; Huntley et al. 2014; Scadding et al. 2015).

Physico-chemical provenance techniques may be usefully divided into two broad categories: surface and bulk. Surface techniques are important for quantifying the different constituents of composite materials and describing physico-chemical alterations such as leaching in aqueous or sedimentary environments, and oxidation at the sample-atmosphere interface. Surface techniques characterise the outer layer of a sample, often with minimal preparation of the specimen, although analyses of cross-sections are also common (e.g. by laser ablation, or raster mapping by Scanning Electron Microscopy). Depending on the aperture or optic/beam dimensions, surface analyses are typically undertaken on, and therefore characterise, a circular or ovoid area microns to millimetres in diameter, with similar penetration depths. Examples of surface techniques include chemical (e.g. Laser Ablation Inductively Coupled Plasma Mass Spectrometry, Scanning Electron Microscopy including Energy Dispersive Spectroscopy, X-Ray Fluorescence) and structural analyses (e.g. X-Ray Diffraction, Raman Spectroscopy, and Fourier Transform Infra-Red Spectroscopy) and can incorporate a mapping function to spatially illustrate features of interest. A range of field-portable surface analytic instruments have become commercially available in recent years, including for Raman Spectroscopy, Fourier Transform Infra-Red Spectroscopy, portable X-Ray Fluorescence (pXRF), and portable X-Ray Diffraction.

Bulk techniques have been preferred in provenance investigations as they dilute mineral segregation[1] and other taphonomic effects[2] (Glasscock & Neff 2003). Bulk analytics include sourcing study staples such as Neutron Activation Analysis, Inductively Coupled Plasma Spectrometry of chemically digested materials using Atomic Emission Spectroscopy, Optical Emission Spectrometry or Mass Spectrometry, and X-ray (powder) Diffraction. Bulk techniques generate data from an area volume of sample, characterising the whole rather than constituent minerals or grains. Bulk techniques are generally performed on extracted samples that are prepared via mechanical grinding into powder or acid digestion, and sometimes via column chemistry using solvents to separate out constituent fractions for analysis (Moyo et al., 2016).

Sourcing studies are the same as other archaeological specialities in that they apply a set of techniques (the mechanics of the approach) and conceptual frameworks that inform research designs and data interpretations. When conducting archaeometric research, including provenance investigations, researchers should be mindful of their requirement to resolve the topic. For each instrumental application, the researcher needs ask whether the intended approach can suitably address the question at hand (Plog 1982). As noted in Section 1 of this Element, a scientific technique should not be applied because one *can*, but because one *should* (to obtain the kind of information required by the question asked). In this vein, in addition to the usual parameters considered in archaeological science, such as instrument *precision*, *accuracy* and *sensitivity*, the concept of *validity* addresses whether the physico-chemical attributes measured in fact relate in a meaningful way to the archaeological phenomena under study (Frahm 2012: 170). Validity is central to provenance research, as 'characterisation requires a bridge between analytical measurements (elemental concentrations, isotope ratios, proportions of mineralogical phases, etc.) and the archaeological concept of

[1] The difference in the composition of the outer surface of artefacts/materials and their bulk composition, which forms at the boundary between the specimen and its environment (Malainey 2011: 171).
[2] Noting that most analysts will remove the outer surfaces of materials before sampling, for instance burring off the upper few millimetres of an ochre surface before drilling powder for analysis.

interest' (Neff 1998: 323). In other words, the characteristic composition and/or structure being physico-chemically measured needs to be relevant to the archaeological problem under investigation—for the purposes of this Element, archaeological landscapes. Yet the concept of *validity applies equally to the selection of the materials for study* as it does to the techniques used to analyse them.

The most important decisions directing data collection, especially in sourcing studies, is where to look. Consideration of scale is essential when selecting assemblages for study. Scale is at the heart of human mobility, in particular trade and exchange networks that are the predominant subject of sourcing studies. For instance, 'customary exchange' as embedded in social networks is often (but not always) thought of by archaeologists as a hand-to-hand activity between individuals, whereas 'trade' operates at a larger scale where the exchange of goods happens between cultural groups and may be part of dedicated trade routes, including down-the-line exchange where material culture and valued raw materials pass through multiple cultural groups (Oka & Kusimba 2008).

Portable XRF (pXRF) has been applied in archaeometry for more than 50 years (Guilherme et al. 2008: 444), with recent decades seeing an explosion in use of the technique in anthropology-related disciplines, particularly provenance investigations (Aloupi et al. 2000; Forster et al. 2011; Johnson 2014; Martindale Johnson et al. 2023; Nazaroff et al. 2009; Shackley 2002, 2005, 2021; Sheppard et al. 2010; Speakman et al. 2011: 3483). In recent years, the ubiquity of pXRF spectrometers has been driven by an increase in the commercial availability and associated decrease in price of 'hand-held' instrumentation, colliding with the growing importance of so-called 'non-destructive' methods in contemporary archaeology (Frahm & Doonan 2013; Heginbotham et al. 2010: 178; Joyce 2011: 199), and targeted marketing/product development by some manufactures to the archaeometry sector. Perhaps like no technique before it, the rapid adoption of commercially manufactured, hand-held pXRF spectrometers has left an indelible impact on archaeological sourcing research due to accessibility.

Historically constrained by the expense of large analytic programmes and a dearth of readily available archaeological scientific expertise, the unprecedented sampling freedom afforded by field-portable, non-invasive spectrometry has facilitated almost unrestricted sampling opportunities. A danger with field-based (including museum) spectrographic techniques is that data collection can degenerate into opportunism rather than focused research design. Before data collection, it is vital to critically consider what cultural phenomena are being investigated to ensure the materials selected for analysis can address it. Again, and this is worth repeating given its overwhelming importance, like Anita Quiles's advice about radiocarbon dating cited in Section 1 of this Element: with regards to physico-chemical characterisations, including sourcing studies, especially those utilising portable analytic instruments, just because you *can* collect a lot of data, does not always mean that you *should*. Combining archaeologically meaningless data with the purposeful analysis of material culture and raw materials used in their manufacture risks diluting or derailing insights into past human societies. At their worst such ventures are little more than meaningless masquerades of scientific number-gathering at the expense of well-considered, meaningful research. Similarly, analysts should select the right tool for the job. Another 'trap' archaeometrists have fallen into with pXRF and other portable instruments, particularly among novices but also among more senior researchers with glitter in their eyes at the thought of obtaining numerical data with advanced technology, is seeing such scientific instruments as the preferred analytics due to their accessibility—having a pXRF spectrometer makes everything look like a good target for chemical characterisation, just as having a hammer makes everything look like a nail. But there is no point in using a hammer if you need a screwdriver.

Archaeometric practitioners need to be aware of, and honest about, the quality of the data they have generated, and the bearing this has on the confidence of their interpretations. Further important considerations when selecting samples and applying portable analytic techniques encompass the (re)introduction of complex physical interactions that are generally accounted for in laboratory settings. Portable instruments are most often used to analyse unprepared materials in archaeological contexts, especially for field and museum-based studies. Unprepared specimens can (re)introduce complexities into analyses that dilute the resolution (accuracy and reliability) of data, something which can be accounted for by sample preparations (for recent examples, see Marino et al. 2022) and by the sensitivity of laboratory instrument settings (i.e. highly stable environments).

Provenance investigations rely on the comparison of compositional data, commonly chemical concentrations. It is true that portable commercial instruments, such as pXRF, were designed to cater for the mining and metals recycling industries, and therefore, to be 'black boxes' in regard to data generation, for use by scientific novices outside of a laboratory environment (Speakman & Shackley 2013). It is also true that ultimately the user is responsible for accuracy and reproducibility, and any problems converting the spectra to comparable semi-quantitative or quantitative data lie with the user, not the manufacturer (Speakman & Shackley 2013). As predicted, some archaeometric users of pXRF with varying scientific backgrounds have gained sophisticated proficiency in calibrating widely sharable data (Frahm 2013; Martindale Johnson et al. 2023). However, varying competencies remain a perennial issue within the archaeometric community, and the importance of our ability to combine datasets and the pitfalls of 'siloed' results that are only internally consistent to specific studies, and/or to particular instrumentation, have been hotly debated in provenance research and remains topical with the continued uptake of affordable geochemical techniques like pXRF by archaeologists (e.g. see Frahm 2013; Shackley 2010; Speakman & Shackley 2013). External calibration is needed to combine datasets (Glascock 2020; Killick 2015; Martindale Johnson et al. 2023; Speakman & Shackley 2013). There is, however, no impediment to comparing data trends, nor testing of broad-scale patterning across regional assemblages (Frahm 2013; Huntley et al. 2021), but the limitations of such qualitative approaches must be acknowledged.

Recognising potential pitfalls in no way detracts from the fact that portable spectrographic techniques can be extremely useful for archaeological applications. An obvious example is the widespread adoption of pXRF for examining archaeological obsidian, largely because the characteristics of the raw material itself are ideal. Chemical variation between obsidian sources is usually 'truly huge' compared to within-source variability (Neff 1998: 324). The suite of elements reliably measured by commercially produced pXRF analysers sits right in the 'sweet spot' of the discriminant elements that differentiate obsidian sources (Speakman & Shackley 2013). That is, XRF, including pXRF, has optimal accuracy in mid-Z elements known to discriminate between obsidian flows (Craig et al. 2007; Nazaroff et al. 2009; Shackley 2002, 2005; Sheppard et al. 2010). Finally, obsidian is as an exemplar material for XRF because it is texturally uniform and consistently dense. Added to these advantages, energy dispersive XRF (including pXRF) only requires corrections for the normal drift of the signal, carried out with a reference sample, once calibration curves are constructed (Ghidotti et al. 2021; Martindale Johnson et al. 2023).

Archaeologists have a responsibility to understand the fundamentals of any technique, scientific or otherwise, that may extract the maximum information from the material remains of past cultures (Lougoute 1982). That way at least if they cannot do it themselves, they will be in a position to connect with those who do, and importantly to evaluate any results they are provided. The historic dearth of archaeological scientific expertise and high cost of analyses has limited the number of specimens physico-chemically characterised, leading to a focus on the exotic. That is, faced with limited opportunities for physico-chemical analyses, understandably most archaeologists have hitherto selected materials that appear exotic to establish if long-range social interactions were occurring (e.g. Smith et al. 1998). The cultural importance of goods and materials may have been overstated in research designs that have sought the exotic and built narratives upon cultural exchange, social interaction and the long-distance diffusion of goods and ideas (e.g. Brumm 2010; McBryde, 1987, 1997a, 1997b, 2000; Mulvaney 1976). The availability and take-up of portable analytic techniques such as pXRF has facilitated the collection of large datasets, resulting in more nuanced understandings of the local signatures of raw materials used by past communities alongside exotics (Barberena et al. 2019; Huntley et al. 2021; Mills et al. 2022), contributing important insights into the complex human-landscape relations of past societies.

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