



Chemical analysis of metalwork and metalworking debris

The composition of artefacts or samples can be determined by chemical analysis. The method of analysis chosen depends mainly on the answers needed. Some types of chemical analysis are quantitative, providing precise information about composition in percentages or parts per million; others give qualitative results, identifying the main elements or compounds present, and provide a rough idea of relative concentrations. Some methods require samples which will be destroyed by the analysis (destructive analysis) but surface analysis can be performed without damage to the artefact. Other considerations are cost and availability of equipment.

Analyses are undertaken for a number of reasons. With metalworking debris, the analysis can identify the metal being worked or the specific process being carried out. With metal objects the composition of the bulk metal or of an inlay or plating can be an aid to accurate description. A group of related artefacts can be analysed to show patterns of alloy use. Distinctive trace element 'fingerprints' may suggest a provenance for the artefact or for the metal of which it is made.

The most commonly used method of non-destructive

analysis is **energy dispersive X-ray fluorescence (XRF)**. Major, minor and some trace elements can be detected. Analysis in a vacuum allows the detection of some lighter elements (down to sodium), but oxygen, carbon etc cannot be detected. The technique is widely used in confirming alloy identifications (eg bronze, brass) and coatings (eg gilding, silvering and tinning on ferrous and non-ferrous metals). Quantitative XRF analysis requires sampling or surface preparation.

A scanning electron microscope (SEM) with an attached **energy-dispersive X-ray analysis (EDX)** system is a convenient method of analysis. Using the SEM image, the area of analysis can be pinpointed exactly, avoiding unwanted areas, eg corrosion products. Samples are normally cut or sawn from artefacts (as for metallography), then mounted and polished, but whole artefacts may be analysed in this way, if the artefacts are metallic and robust. For most elements as little as 0.2% can be detected. Light elements down to sodium or to carbon (depending on the machine being used) can be analysed for. Fully quantitative analyses can be provided.

Wavelength-dispersive X-ray

analysis (WDX) gives similar results, but the detection limits are lower so it can be used to analyse elements present at very low concentrations (less than 0.2%). WDX can either be a stand-alone system or can be attached to an SEM. A microprobe is similar to an SEM with WDX but the machine is designed to optimise the analytical function with imaging as a secondary consideration.

Samples for all these quantitative X-ray methods can be very small (1-2 mm across) and the area analysed can be far smaller than this. The sample remains intact and may be re-analysed later.

Small (c 10mg) metal samples cut or drilled from artefacts can be analysed using **inductively-coupled plasma spectroscopy (ICP)**. The samples are totally consumed in analysis. A wide range of elements can be determined simultaneously. Detection limits are normally very low, unless the sample size is unusually small. Information about sample structure (eg casting history) cannot be recovered by this technique.

Atomic absorption spectroscopy (AAS) was the precursor to ICP and is still used in many laboratories. It is relatively time consuming as each

element must be analysed for separately. If a large number of elements are analysed for this makes the technique expensive, but the machines themselves are relatively cheap and widely available.

The above methods all identify the elements present in an artefact or sample. Other analytical techniques provide other information.

Crystalline compounds (*eg* corrosion products) can be identified using X-ray diffraction (XRD). Very small samples (about the size of a pin head) can be analysed, although the best results are usually from larger samples. Where several compounds are present in one sample, it may be difficult to distinguish all of them.

Lead isotope analysis can be performed on artefacts containing even small mounts of lead. The sample size required depends on the amount of lead present, but in lead-rich materials can be a few milligrams. The isotopic characteristics of the sample relate to the geological origin of the metal, but they can be confused by alloying. A large number of reference samples (of known geological origins) are required for accurate sourcing.

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