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NEWS

New Analytical Equipment Expands the Capabilities of the Institute's Laboratories

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The characterisation of materials is an important aspect of much of the scientific and conservation work at the Institute, and the expansion of our facilities to include X-ray diffraction (XRD) and infrared spectrometry (FTIR) is very welcome. The new equipment (located next to the conservation laboratory on the sixth floor) was acquired this academic year through the College from several grant applications by Professor Ian Freestone, Dr. John Merkel, James Hales and other staff. The new equipment extends our analytical capabilities beyond the current techniques for chemical analysis and imaging in the Wolfson Archaeological Science Laboratories, to the identification of compounds or minerals. This allows more rigorous and precise understanding of the make-up of an artefact.

The improved analytical capabilities allow new questions to be addressed, leading to new interpretations of the archaeological evidence. Furthermore, they allow the investigation of past environmental conditions and deterioration, which contributes to the selection of appropriate conservation options for artefacts under treatment in the Conservation Laboratory. XRD and FTIR are also useful in determining the causes of active deterioration and corrosion of artefacts in storage or on display.

The XRD is a Rigaku Miniflex 600, a small unit which sits on a laboratory bench. Its user-friendly software can access databases which match diffraction patterns and aid identification. While it has a wide range of applications, in general XRD is used most frequently to identify mineralogical or inorganic constituents of materials. A small amount of sample, typically about 0.1 g, is crushed and ground to powder for analysis. The FTIR, a PerkinElmer Spectrum Two IR Spectrometer, is also a small bench-top instrument. Infrared spectroscopy is primarily designed to provide qualitative molecular analysis of a wide range of organic materials and is an excellent partner technique to the many methods of elemental analysis we have currently at the Institute. The new equipment has a number of interchangeable sample modules and allows traditional transmitted spectroscopy to be undertaken as well as Diffuse Reflectance and Attenuated Total Reflectance spectroscopy (for which minimal to no sample preparation is needed).

Students are trained in the use and application of XRD and FTIR during courses in Conservation and Early Technology and Materials. Experience with these techniques for materials identification allows students to develop their own abilities and confidence through hands-on technical studies of archaeological artefacts. Several hundred samples have been identified so far this academic year. A few examples below represent the range of projects undertaken.

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The analysis of pigments is a research topic well-suited to investigation with XRD. Many inorganic pigments used in antiquity have been deliberately mixed with other materials to 'adjust' the final colour. For example, a red pigment can be mixed with different proportions of gypsum, calcite or quartz to obtain different shades, from red to pink. Using XRD both the components and their proportions can be determined. We have recently examined the partially degraded red pigment found on a pre-Hispanic gold funerary mask excavated at the Sican site of Batan Grande in northern Peru (Shimada and Montenegro 1995). XRD enabled us to detect both cinnabar (mercuric oxide) a well-known red pigment, but also malachite (copper carbonate). Malachite is a common corrosion product of copper and, in this case, is not a deliberate addition but derives from the underlying mask which is a complex gold-silver-copper composition. The malachite helps to characterise the burial environment, but it now darkens the colour of the original red-cinnabar pigment used to decorate the mask, which would have appeared significantly brighter when it was originally used.

The Institute has strong interests in early metallurgy and its evolution. Research on the very early production of copper in Serbia recovered a range of distinctively coloured minerals from excavations and in nearby mines (Radivojević *et al.*, 2010). XRD identified these as complex tin-containing copper-rich minerals. These unusual coloured minerals were apparently selected deliberately by the early metal workers to produce 'natural' alloys of copper and tin which had more desirable properties than pure copper (Radivojević *et al.*, 2013).

As part of monitoring for active corrosion on archaeological metal work in museum stores or on display, small samples can be collected and identified with XRD. For example, 'bronze disease' observed as a powdery, light green corrosion product can be identified more precisely as the mineral paratacamite ($Cu_2(OH)_3Cl$). Likewise, copper acetates and copper formates can be identified and attributed to deterioration due to emissions from organic storage materials such as wooden cases or shelving (Paterakis *et al*, 2011). In another recent application, the mineral akaganeite (β-FeOOH) was identified as corrosion on iron. The identification of these different minerals as indicating active corrosion is an important aid in making decisions about appropriate conservation treatments (Scott, 2002; Scott and Eggert 2009).

Current research into the effects of bat urine and droppings on church furnishings (reported elsewhere in this issue) has recently employed both FTIR and XRD in preliminary analysis of bat droppings and the corrosion products caused by bat droppings and urine on various building and decorative materials. Bats frequently roost in ancient churches and roost sites are protected in Britain (Howard, 2009), so precise identification of the corrosion products and other chemical reactions is critical to understanding and addressing the effects of these bat colonies.

Students have been using FTIR to identify unknown materials and residues found on artifacts, such as waxes, based on comparison with known reference spectra. A recent project used FTIR to identify material found in the Honor Frost Archive and labelled mysteriously as 'fish poison from the Lebanon'. The contents of the package were ultimately identified with the help of the Royal Botanic Gardens, Kew as coming from the storax plant (*Styrax officinalis*) which was indeed traditionally used as a fish poison (Hepper 2004).

Conclusions: advances in examination

On BBC iPlayer, it is now possible to watch Sir Mortimer Wheeler (founding Director of this Institute) and other scholars in the early television programme 'Animal, vegetable, mineral?' identifying unusual objects. The first programme was broadcast in 1954 and, among a variety of objects, included a Roman

Fig. 1: The Lyminge coulter on display in Maidstone Museum (Photo: John Merkel).

iron plough coulter from Bigbury Camp, Kent. It was successfully identified as iron, but the mineral constituents on the surface seemed unremarkable. Identification of minerals, such as different iron oxides, was then based upon visual observations and long experience alone.

Today we can make more precise mineral identifications. Very recently another iron coulter, of a later type, from Lyminge, Kent, excavated by Dr. Gabor Thomas of the University of Reading (Thomas and Knox 2012), was examined at this Institute using XRD. This provided much more detailed understanding of the corrosion, and showed that the coulter had a stable layer of magnetite (Fe_3O_4) protecting the surface. The presence of magnetite provided new information about the original burial conditions as certain minerals form and persist only under unusual environmental conditions. The layer of magnetite also best represented the original shape, dimensions and signs of use-wear on the coulter (which were revealed by investigative cleaning of the overlying adhering soil and other corrosion products). After conservation, the iron coulter was recently installed in a temporary exhibition on the archaeology of Lyminge at the Maidstone Museum (**Fig. 1**).

The acquisition of these new XRD and FTIR instruments significantly expands our capabilities in the area of materials characterisation. Post-graduate archaeology students and staff now have much greater access to this type of analytical equipment for application in a wide variety of research projects. Accurate and precise identifications of both inorganic and organic materials are fundamental for technical studies in archaeological conservation. In turn, the new analytical equipment enables a more co-ordinated and interdisciplinary approach to study of archaeological materials.

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