

**Scientific services
for cultural heritage
and archaeology**

Sydney
Analytical

THE UNIVERSITY OF
SYDNEY



“Excellence in research is increasingly linked to excellence in capability, and that is what our core research facility program is all about.”

Professor Simon Ringer
Director Core Research Facilities

Sydney Analytical

Who we are



Sydney Analytical is a multidisciplinary facility supporting research excellence across the University of Sydney. Its state-of-the-art research infrastructure supports research and development in fields spanning: nanoscience; clinical medicine; medical and life sciences; chemistry; physics; agricultural, environmental and veterinary sciences; engineering; pharmaceutical sciences; geosciences; and museum and cultural studies.

Sydney Analytical also engages extensively with industry, including a collaboration with ANSTO to support neutron and synchrotron research.

Included in the range of instrumentation held by Sydney Analytical are modern and non-destructive analytical techniques that enable new insights to be gained from items in museum and art gallery collections, as well as archaeological and palaeontological artefacts.

Chemical and morphological analysis is making important contributions to research problems in archaeology, palaeontology, art, collection conservation and many other areas.

Professor Peter Lay
Academic Director
Sydney Analytical
Core Research Facilities

Cover art: Artist unknown, Madonna and Child with saints and angels, c.1450-60, donated through the Hon. RP Meagher bequest 2011 (UA2012.688), courtesy of the University Art Gallery and Art Collections, Sydney University Museums. This icon appears to date to the Renaissance and was analysed at Sydney Analytical to identify pigments using infrared spectroscopy.

A hub for science in cultural heritage and archaeology

The use of analytical chemistry in cultural heritage and archaeology is a powerful means of investigating a wide array of important issues through the analysis of materials from the biological to inorganic.

Sydney Analytical houses more than 30 high-end instruments and spans multiple vibrational spectroscopic and X-ray techniques, many of which are the most advanced of their kind in Australia and can provide non-destructive, rapid and contactless analysis.

Analysis of photographic film using portable ALPHA FTIR spectrometer, equipped with ATR accessory





Contactless NIR analysis of pigments on the cedar coffin of the woman Mer-Neith-it-es; 26th Dynasty (664 BC to 525 BC, Saite Period); Saqqara, Lower Egypt (NMR.29.1-2) Nicholson Museum, the University of Sydney

Common materials

- pigments, binders, inks and paints
- varnishes
- bone, ivory and teeth
- textiles and manuscripts
- ceramics, glazes and glass
- photographic film
- residues
- plaster.

Common questions

- dating
- material identification
- conservation
- provenance
- degradation
- production processes
- fraud identification.

Our facilities

Our range of instrumentation covers not only laboratory-based equipment but also portable instruments.

Portable instrumentation allows on-site non-destructive analyses in situations where sample location, access, size or fragility make it otherwise impossible to perform laboratory-based investigations, particularly important to museum and art gallery collections, and archaeological studies in the field.

Our technical staff are available to travel to conduct this type of on-site research, while experienced users may request extended and unsupervised use of instrumentation.

Vibrational spectroscopy

- Infrared spectroscopy
- Raman spectroscopy
- Macro → Nanocapabilities
- 2D and 3D mapping, imaging, depth profiling
- Controlled-environment techniques

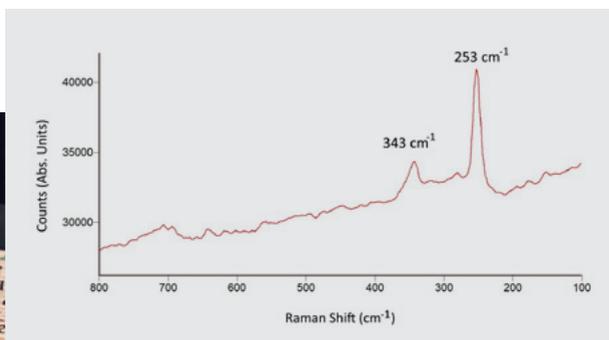
X-ray techniques

- Powder diffraction and single crystal
- Small-angle X-ray scattering
- X-ray/ultraviolet photoelectron spectroscopy
- X-ray fluorescence spectroscopy (point and mapping)
- Controlled-environment techniques

What is vibrational spectroscopy?

Vibrational spectroscopy encompasses infrared and Raman spectroscopy. Both techniques can be used to determine what compounds are present in specimens of interest. Vibrational spectroscopy can be applied to a wide variety of materials, from biological to inorganic. Commonly analysed materials include pigments, binders, inks, paints, varnishes, residues, textiles, manuscripts, ceramics, glazes, glass, bone, ivory, teeth, plaster and photographic films.

In infrared spectroscopy, the sample is irradiated with polychromatic light and a photon of light is absorbed when the frequency (energy) of the absorbed light matches the energy required for a particular bond to vibrate within the sample. In order for a vibration to be infrared active, the molecular dipole moment must change during the vibration. Infrared spectroscopy is useful for inorganic and organic specimens, particularly valuable in the analysis of the latter.



Raman spectrum of vermilion (above) in a medieval manuscript (left)

In Raman spectroscopy, the sample is irradiated with monochromatic light and the photons are either inelastically or elastically scattered. The inelastically scattered light (Raman scatter) has lost (Stokes) or gained (Anti-Stokes) energy during this interaction and the emitted photon contains information about the molecular structure of the sample. The elastically scattered light has the same energy as the incident laser light and is called Rayleigh scatter.

Modern Raman instruments are designed to filter out the Rayleigh light, as only one in every million photons will be Raman scattered. For compounds to be Raman active, when the molecule vibrates there must also be a change in polarisability, ie, a change in the shape, size or orientation of the electron cloud that surrounds the molecules.



Bruker Tensor 27 FTIR spectrometer with Hyperion 3000 microscope



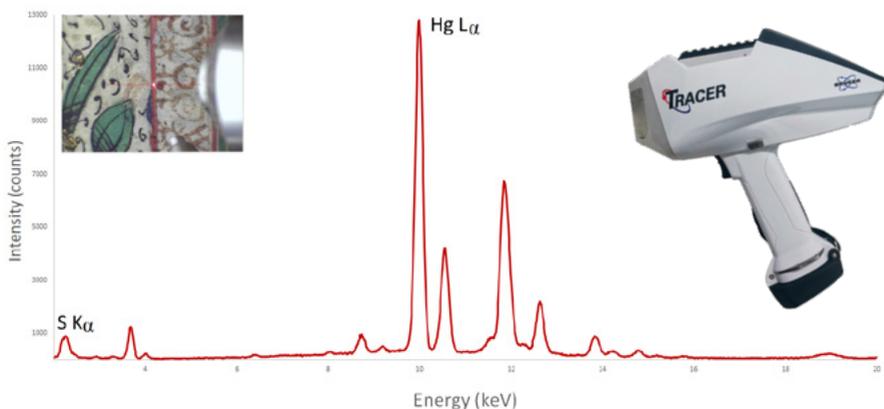
Renishaw InVia Dual Raman System with inverted and upright microscopes

What is X-ray fluorescence spectroscopy?

X-ray fluorescence spectroscopy (XRF) enables the identification of elements present in samples and their quantification. In XRF, an X-ray source irradiates the sample and if an incoming X-ray has enough energy it can eject an electron from its shell in an atom. This leaves a vacancy in the shell that is filled by a higher energy electron. When this happens, an X-ray photon can be emitted. The energies of these X-ray photons depend on the shells involved in each atom, which are specific to each element. As such, elements in the sample emit X-rays of discrete energies

that are characteristic for the element allowing identification of the elemental composition of samples.

XRF can be applied to a wide variety of sample types, including solids, liquids and powders over a wide range of material types, organic and inorganic. Depending on the application, XRF analysis can be fast, require minimal to no sample preparation and be non-destructive. Elements from sodium (Na) through to uranium (U) are typically identifiable. Portable instruments have the added advantage of being able to analyse large objects non-destructively.



XRF spectrum of vermilion (HgS) in a medieval manuscript obtained with the ARTAX micro-XRF spectrometer (left), and Bruker TRACER 5i spectrometer (above)

Case studies

Ming porcelain

Raman spectroscopic analysis was carried out on Ming period porcelain sherds recovered from two 17th century Portuguese shipwrecks: the *Santo Espirito* (1608) and the *Santa Maria Madre de Deus* (1643). Analysis showed that two of four sherds from the *Santa Maria Madre de Deus* contained anatase and β -wollastonite. This is characteristic of medium-temperature soft-paste porcelains, not typically encountered in the analysis of Ming period porcelain. This work was conducted in collaboration with the University of Pretoria, South Africa, and University of Bradford, UK.



Obsidian sourcing

Obsidian from the Pacific (sources in West New Britain, Manus and Vanuatu) have been analysed and unknowns sourced to their geological origins using portable XRF and laboratory-based and portable Raman spectroscopy, in collaboration with the Australian Museum. The results have shown the viability of these techniques for provenance work and provided insights into trade and social interaction in the region.

Identification of photographic film types

The identification of film types is of enormous benefit to conservation and storage of such collections. In particular, cellulose nitrate is flammable and large collections can pose a significant fire hazard. Both Raman and infrared spectroscopy can be used to identify film types, and the results of analysis can guide conservators on storage and handling.



Digital colour reconstruction of the coffin of Mer-Neith-It-Es

Using portable XRF, Raman, near infrared and infrared spectroscopy, pigments have been identified on the coffin of Mer-Neith-It-Es, in collaboration with Sydney University Museums, Nicholson Collection and the University of Newcastle, Australia. This information is being used to create a digital reconstruction of the coffin, including the original colours as identified through analysis.



Ivory discrimination

Portable Raman spectroscopy using a 1064 nm laser has been used to discriminate between six types of terrestrial and marine mammalian ivories, along with leopard teeth and fake ivories.



These results show the potential for portable Raman spectroscopy as an effective, rapid, non-destructive method for the identification of ivories and fakes in the field. This work was conducted in collaboration with the University of Bradford, UK.

Identification of pigments used in medieval manuscripts

Analysis of a 15th–16th century Augustinian palimpsest manuscript by both XRF and Raman spectroscopy has been able to reveal the pigments used in the text and illuminated letters and borders. These letters and borders were cut and pasted into the manuscript and the identification of pigments can help us better understand if the letters derive from one or multiple sources. Furthermore, in conjunction with other techniques, XRF mapping is being conducted to reveal the content of earlier erased text. This is a collaborative project with the Rare Books Library at the University of Sydney.



Portable infrared equipment

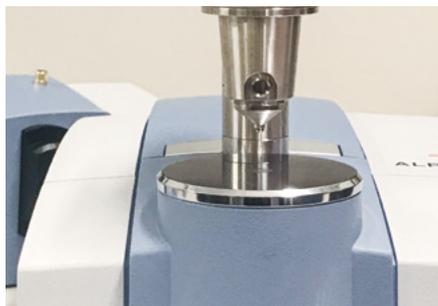


LUMOS FTIR microscope

- Standalone, fully automated, all-in-one microscope and FTIR spectrometer designed to be extremely easy to use while still providing high quality spectra
- Fitted with a $\times 8$ objective capable of measurements in reflectance, transmission and attenuated total reflectance (ATR)
- Digital zoom allows visible light images to be collected at up to $\times 32$ magnification
- Micro-ATR crystal with $100\ \mu\text{m}$ diameter, fitted with an integrated pressure control to ensure consistent application of pressure by the crystal on the sample

Instrument specifications

- Spectral range: 4000 to $600\ \text{cm}^{-1}$
- Spectral resolution: better than $2\ \text{cm}^{-1}$
- Liquid nitrogen cooled MCT detector
- Motorised X-Y-Z stage ($0.1\ \mu\text{m}$ accuracy)
- Objective working distance of $30\ \text{mm}$
- Motorised knife-edge aperture store strict measurement area



ALPHA FTIR spectrometer

- Compact, easy to use FTIR spectrometer
- Wireless communication, battery operation (8 hours)
- No alignment necessary after transportation
- Insensitive to vibrations, allowing the spectrometer to be placed anywhere
- Platinum ATR single reflection diamond for solid and liquid samples
- External Reflection Module (ERM) for non-destructive, contactless analysis of larger samples
- Integrated video camera provides viewing of the sample area

Instrument specifications

- Spectral range: 7500 to $375\ \text{cm}^{-1}$
- Spectral resolution: better than $2\ \text{cm}^{-1}$
- Wavenumber accuracy: $0.01\ \text{cm}^{-1}$
- Room temperature DTGS detector



4300 handheld FTIR spectrometer

- Can be used handheld or mounted on sampling stage
- No alignment necessary after transportation
- Diamond ATR for solid, liquids, pastes and gel samples
- Diffuse reflectance for samples with a matt surface, eg, powders and paper
- External reflectance for analysis of films and coatings on reflective metal surfaces

Instrument specifications

- Spectral range: 4500 to 650 cm^{-1} (DTGS)
- Spectral resolution: 4–16 cm^{-1} (maximum)
- Detector: thermoelectrically cooled DTGS
- Operating temperature: 0 to 50 $^{\circ}\text{C}$ (DTGS)
- Reference sample included for calibration



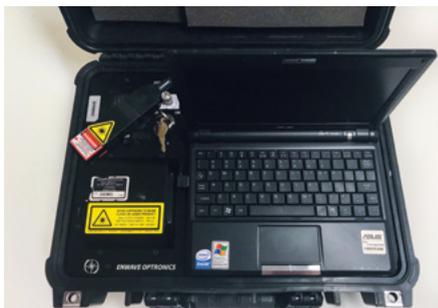
MicroNIR™ NIR spectrometer

- Rugged and durable handheld NIR spectrometer for data collection in the field
- Provides non-destructive analysis of a wide range of samples
- Rapid collection times enable high throughput
- Operated through a tablet or laptop
- Spectra can be directly exported as a data matrix for multivariate analysis in the Unscrambler™

Instrument specifications

- Spectral range: 950 to 1650 nm (10526 to 6060 cm^{-1})
- Detector: 128 pixel InGaAs photodiode array
- Working distance: 0 to 15 mm (3 mm optimal)
- Measurement time: typically 0.25 to 0.5 s
- Operating temperature: -20 to 50 $^{\circ}\text{C}$
- Reference sample included for calibration

Portable Raman equipment



EZRaman Raman spectrometer

- Reliable, compact and easy-to-use Raman spectrometer designed for laboratory or field analysis of samples
- Comes with a robust carry case, laptop computer, optical fibre coupled laser probe; can be run on mains power or via the included rechargeable battery
- Suited for direct, non-destructive analysis of large samples and for any research requiring portability and high sensitivity

Instrument specifications

- Excitation: 785 nm frequency-stabilised laser with maximum output of ~400 mW
- Spectral range: 2000 to 100 cm^{-1}
- Optical resolution: ~4.5 to 6.5 cm^{-1} (average)
- Detector is a TEC-cooled CCD camera (-50 °C), with 16 bit digitisation
- Operating temperature of 10 to 40 °C with thermal shutdown procedure
- Fibre optic probe with 7 mm working distance
- Operates using GRAMS software supporting .txt, .spc, .dat or .bmp files

BRAVO Raman spectrometer

- Reliable high-performance handheld Raman spectrometer for on-site sample identification
- Enables the identification of dark, fluorescing and weak scattering samples
- Uses patented Sequentially Shifted Excitation (SSE™) technology to mitigate fluorescence issues often encountered with Raman analysis
- Two interchangeable sampling heads available for measuring solid and liquid samples
- Data can be stored on the spectrometer or transferred wirelessly to a PC for in-depth analyses and data processing

Instrument specifications

- Excitation: Duo LASER™
- Spectral range: 3200 to 300 cm^{-1}
- Docking station for charging and Ethernet connection to laptop

Portable XRF equipment



ARTAX μ -XRF spectrometer

- State-of-the-art μ -XRF spectrometer that enables examination of large and uneven objects with no sample preparation required
- Performs fast, non-destructive chemical analysis, both spot analyses and mapping, with a spatial resolution of 70 μ m
- Polycapillary lens for beam focusing; extremely high fluorescence intensity reduces measurement time
- Helium flushing

Instrument specifications

- Excitation source: Rh X-ray fine focus tube, maximum 50 kV, 1 mA, 30W; polycapillary lens for micro excitation spot (intensity gain >1000); lateral resolution <100 μ m, for excitation up to Sb K-line
- Detector: Peltier-cooled XFlash® silicon drift detector, 10 mm² active area; energy resolution <150 eV for Mn-K α at 100 kcps; maximum count rate >100 kcps
- Helium purging of excitation and detection paths
- Detects elements from Na to U
- X-Y-Z stage with stepper motors, 50 mm range ARTAX 1D and 2D mapping



TRACER 5i XRF spectrometer

- TRACER 5i can accurately analyse the elemental composition and identify standard or complex materials in the field, with minimal sample preparation required
- Allows complete user control of the excitation conditions which can be developed using recommended settings in the software or user preferences
- Spot analyses with a resolution of approximately 1 cm²

Instrument specifications

- Excitation source: Rh thin window X-ray tube; X-ray generator 6 to 50 kV with 4.5 to 195 μ A, 4 W
- Detector: proprietary 20 mm² silicon drift detector with <140 eV @ 250,000 cps Mn K α
- Detector window: 8 μ m beryllium
- Beam path: capable of vacuum, helium or air to detect elements from F to U
- Collimation: user-changeable collimator; 3 mm and 8 mm collimators supplied
- Filters: 5-position primary beam filter wheel; manual insertion filter/secondary target slot for factory or user-made filters

Contact us

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