

Art and Cultural Heritage applications

Infrared Microspectroscopy
beamline

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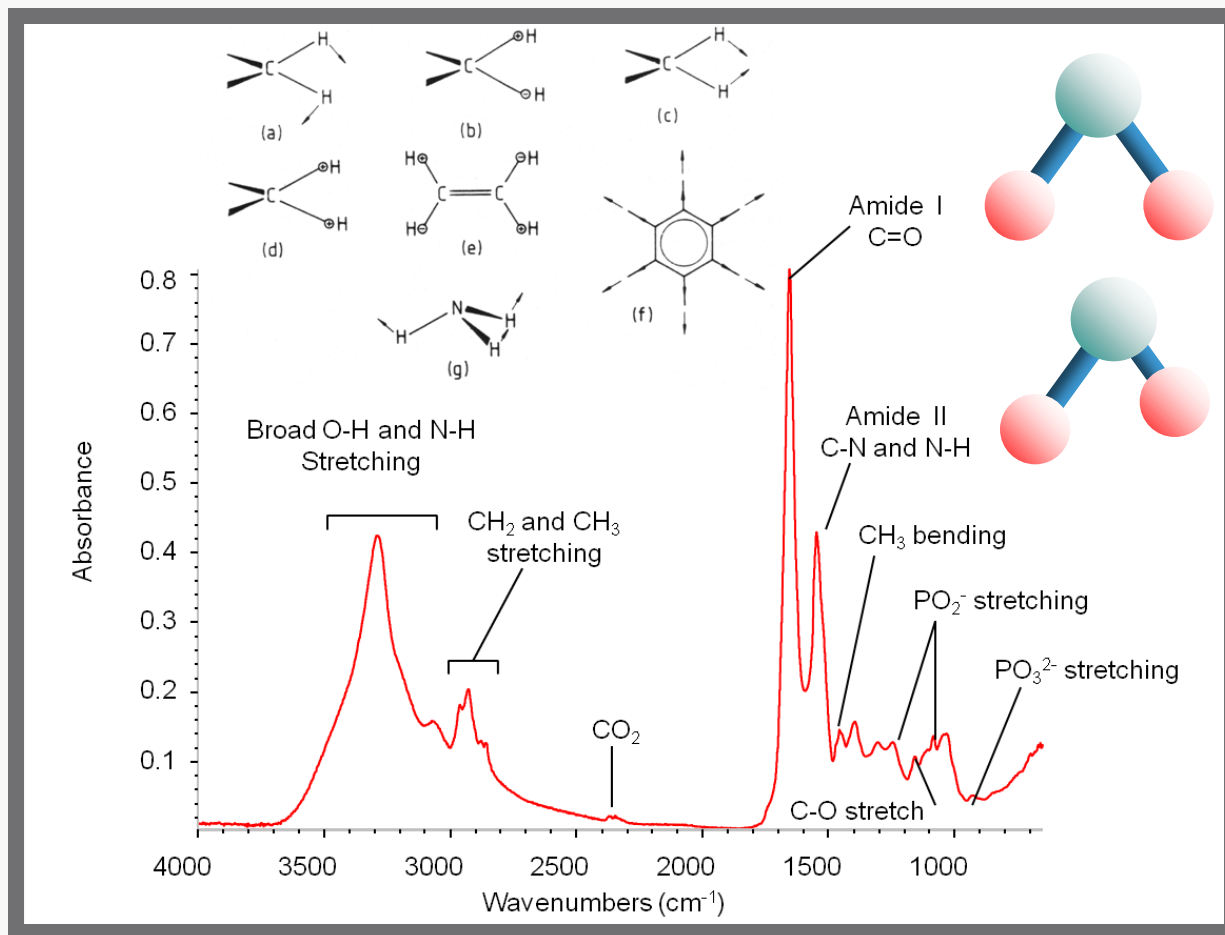
9th September 2010

OVERVIEW

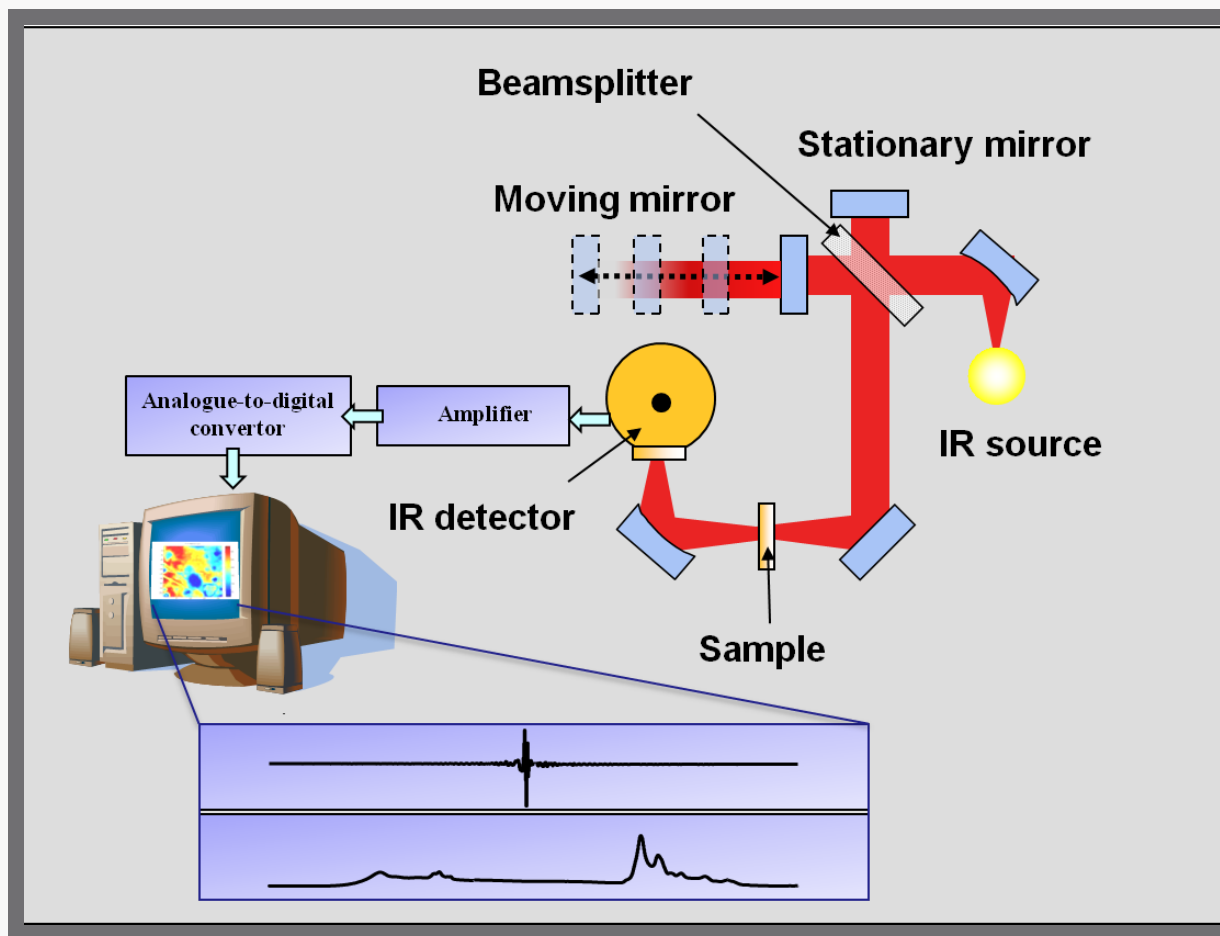


- Introduction to IR spectroscopy and instrumentation
- The advantages of synchrotron-based Fourier-transform infrared microspectroscopy and IR beamline at the Australian Synchrotron
- Methods for measuring IR spectra, sample preparation and data analysis
- Cultural heritage applications at the IR microspectroscopy beamline at the AS

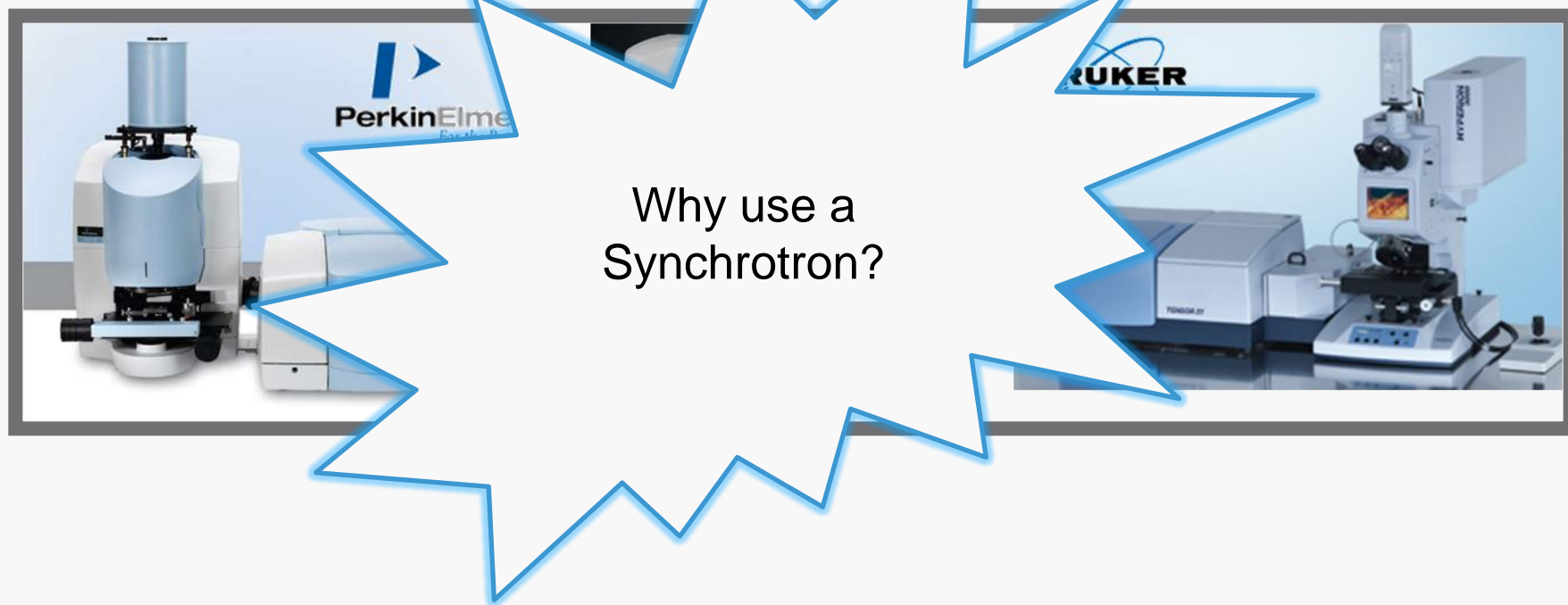
INFRARED SPECTROSCOPY



METHOD OF DATA COLLECTION: FOURIER TRANSFORM INFRARED SPECTROSCOPY

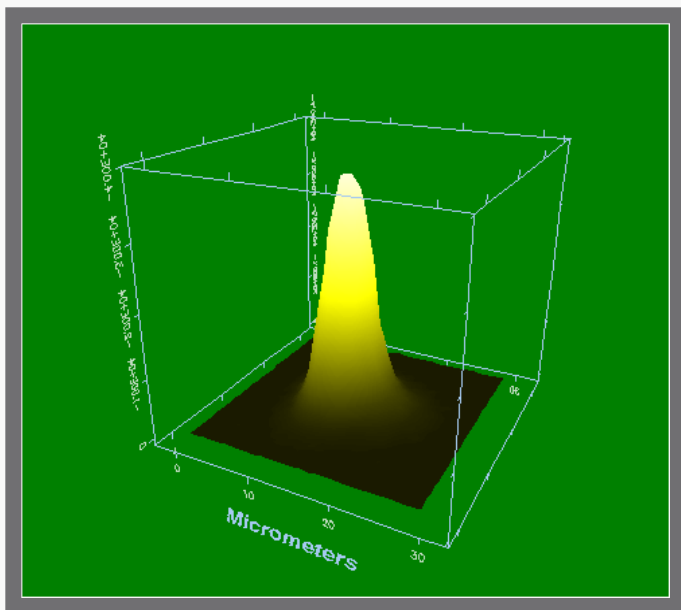


INFRARED MICROSCOPY INSTRUMENTATION



ADVANTAGES OF SYNCHROTRON TO IR MICROSCOPE

- **Brightness 2-3 orders of magnitude higher than a thermal (laboratory based) source**



Beam profile at sample stage:
Microscope IR Beamline at SRS

$$\text{BRIGHTNESS} = B = \frac{P}{\Delta A \cdot \Delta \Omega}$$

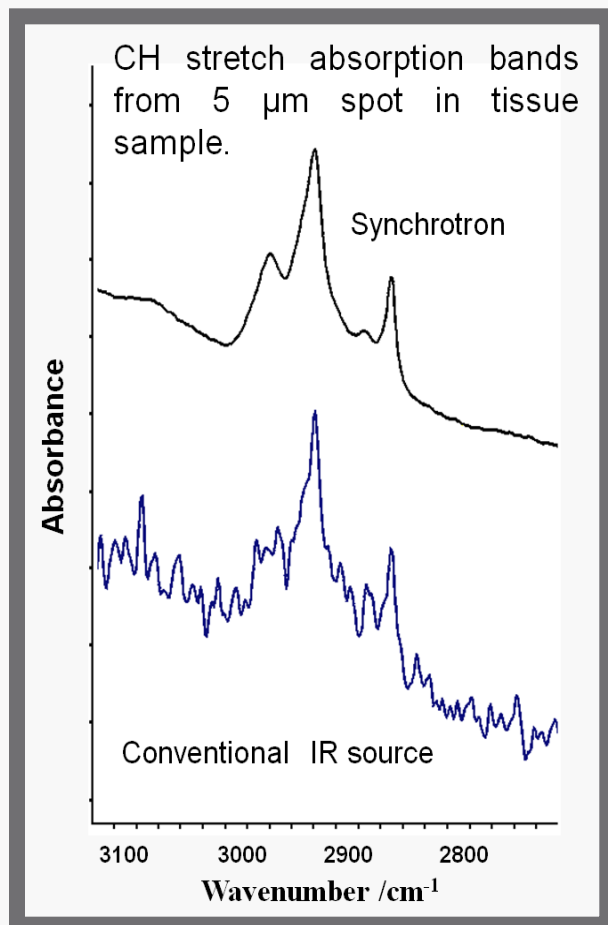
Power per unit area per unit solid angle

Area mapped = 30x30 μm .

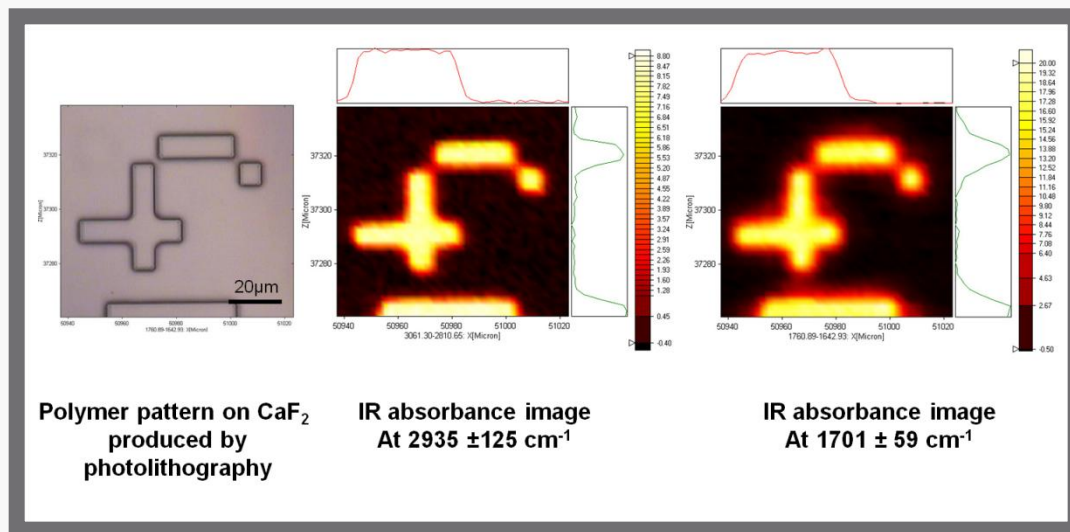
Beam halfwidth = 8x8 μm .

ADVANTAGES OF SYNCHROTRON TO IR

SIGNAL TO NOISE

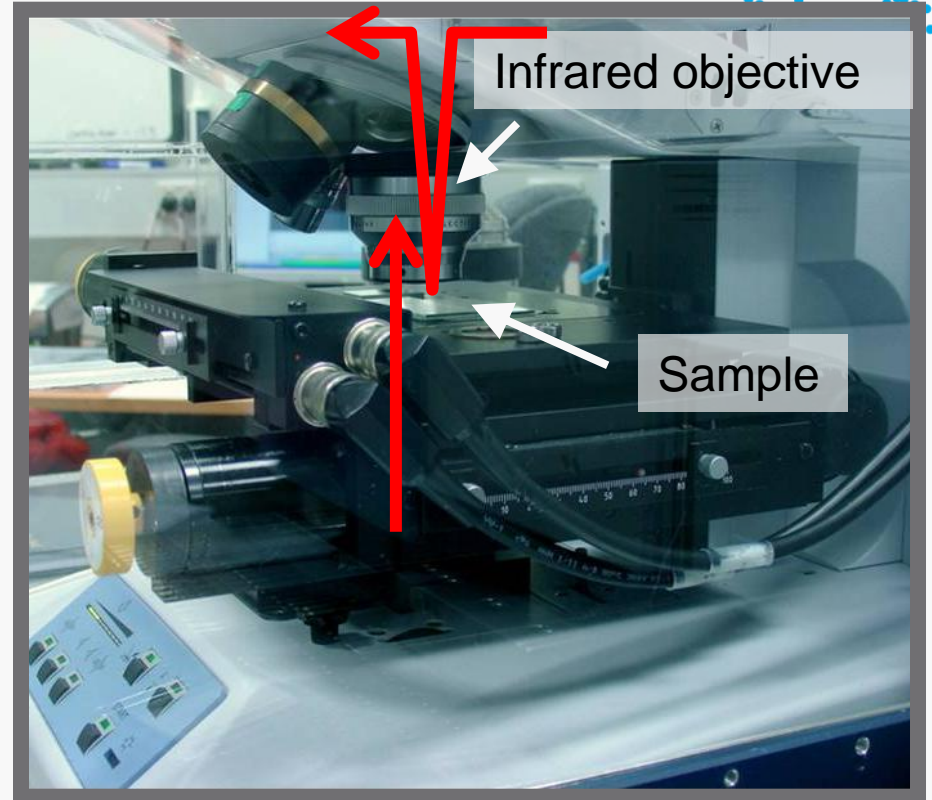


SPATIAL RESOLUTION



- Fast data acquisition with superior signal to noise and enhanced lateral resolution (close to diffraction limit)

INFRARED MICROSCOPE BEAMLINE



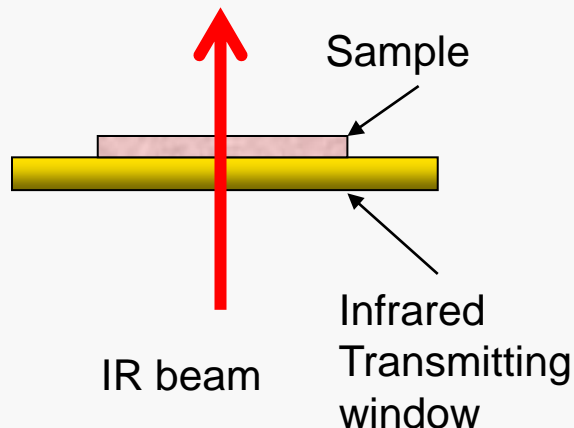
- Bruker HYPERION microscope
- Resolution down to a few microns in Mid-IR

- Computer controlled motorised sample stage
- Bruker OPUS software for data collection and analysis

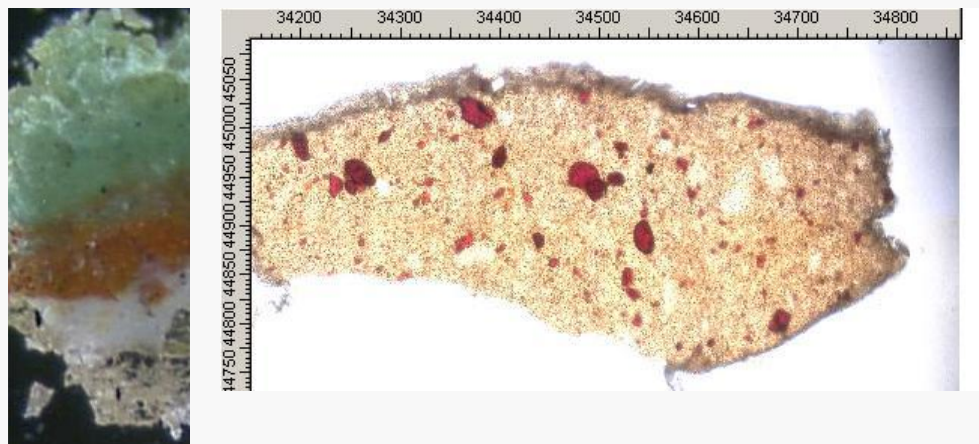
TYPES OF MEASUREMENTS USING IR MICROSCOPE

- Transmission
- Reflectance
- 'Transreflectance'
- Grazing angle Reflectance measurements
- Attenuated Total Reflectance

TRANSMISSION MEASUREMENTS



- Samples should be 10 microns or thinner freestanding, supported or embedded in an IR transmitting material

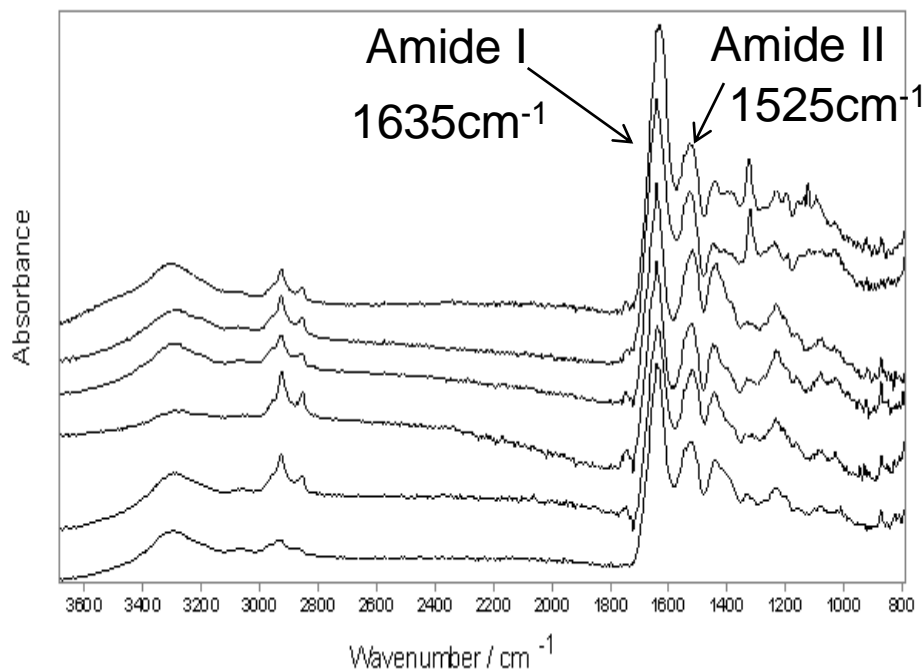


- Sample fragments pressed between diamond windows

DEGRADATION 19TH CENTURY PARCHMET SUPPORTS DUE TO COROSIVE NATURE OF IRON GALL INK



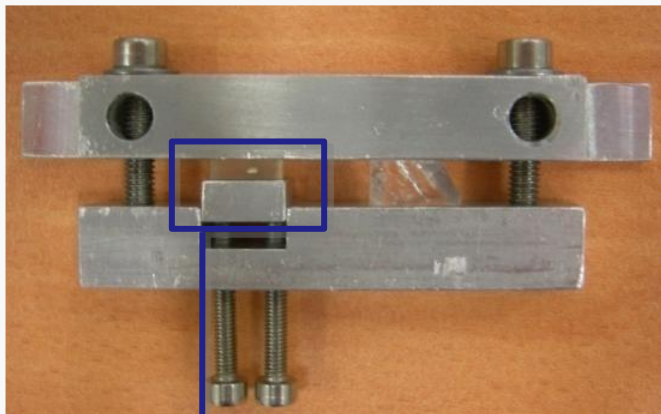
Areas of 19th century indenture parchment document showing iron gall ink corrosion and loss.



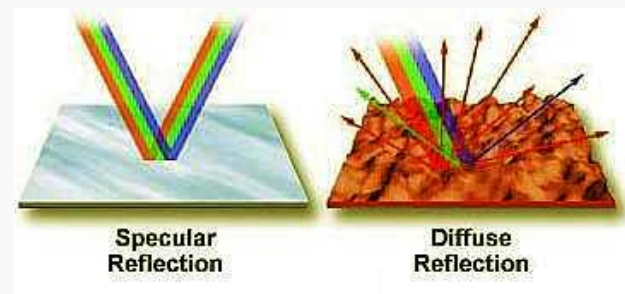
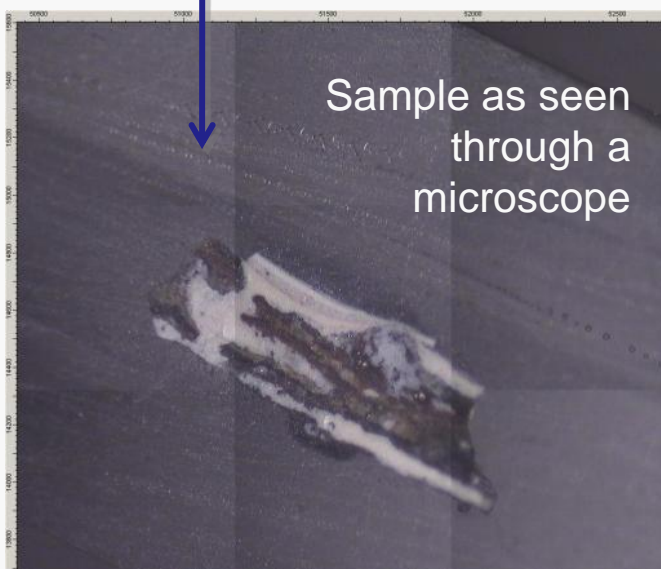
FTIR transmission spectra for a parchment cross-section.

Degradation followed by the changes in Amide bands (information on protein structure) from the collagen present in parchment.

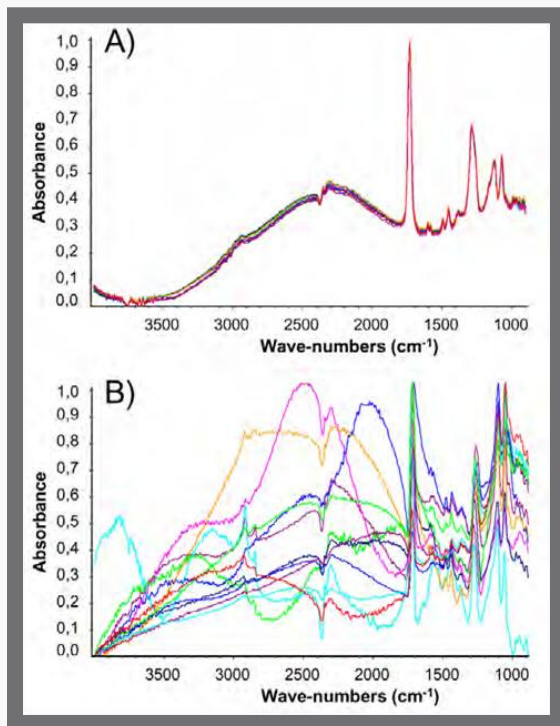
REFLECTANCE MEASUREMENTS: THE PAINT SAMPLE EMBEDDED IN RESIN



- Ideally requires a well polished flat surface
- Spectra require additional correction procedures due to dispersion artefacts (Kramers-Kronig-Transformation).



REFLECTANCE MEASUREMENTS: THE PAINT SAMPLE EMBEDDED IN RESIN



Reproducibility of FTIR spectra acquired in reflection on the surface of a resin block when the surface is obtained:

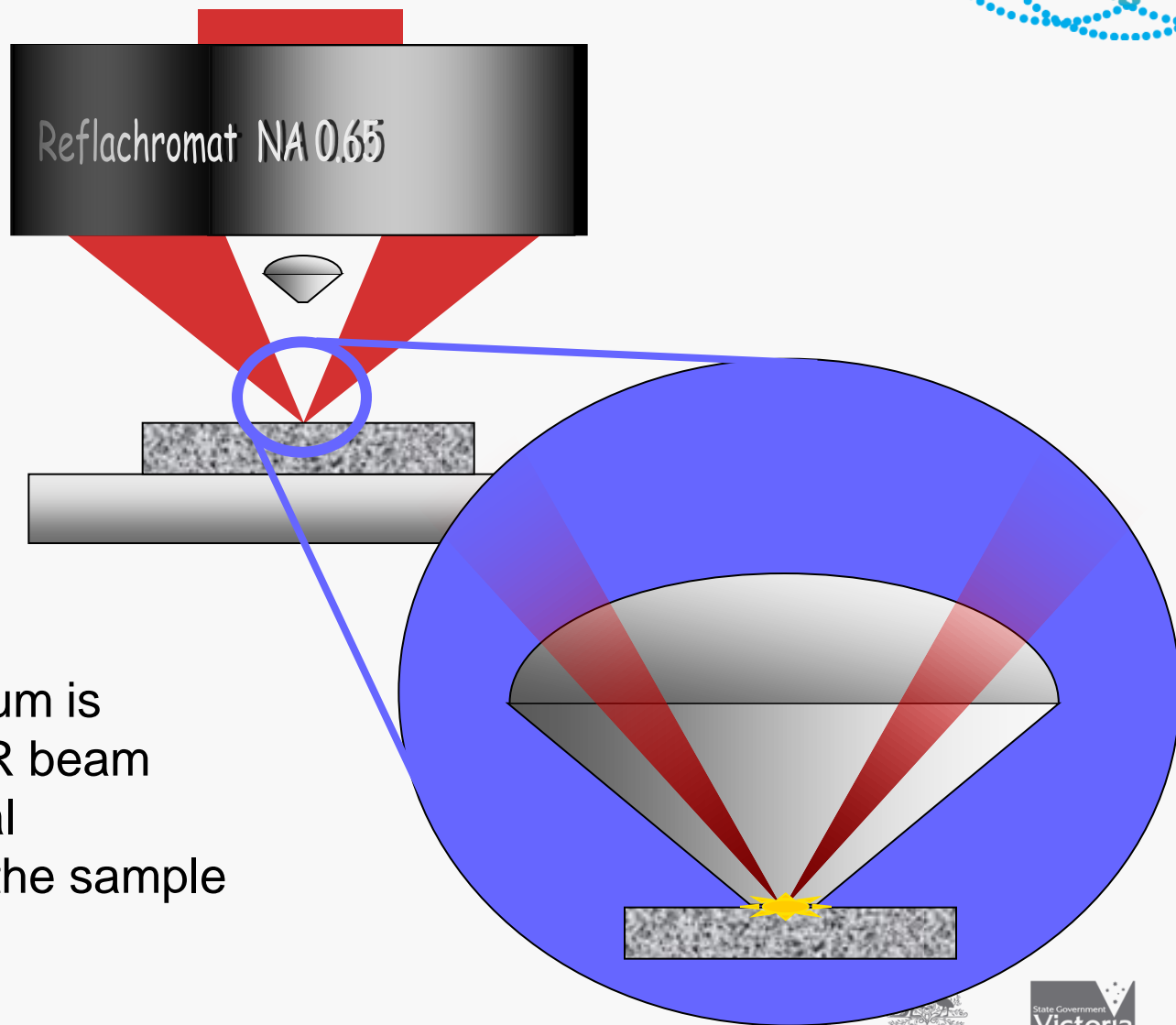
A) by cutting with an ultra-microtome,

B) by polishing with silicon carbide (grade 1200).

Aperture size: 12 12 μm²

Synchrotron-Based Micro-Imaging for Paintings, e-PS, 2009, 6, 1-9

ATTENUATED TOTAL REFLECTION (ATR)

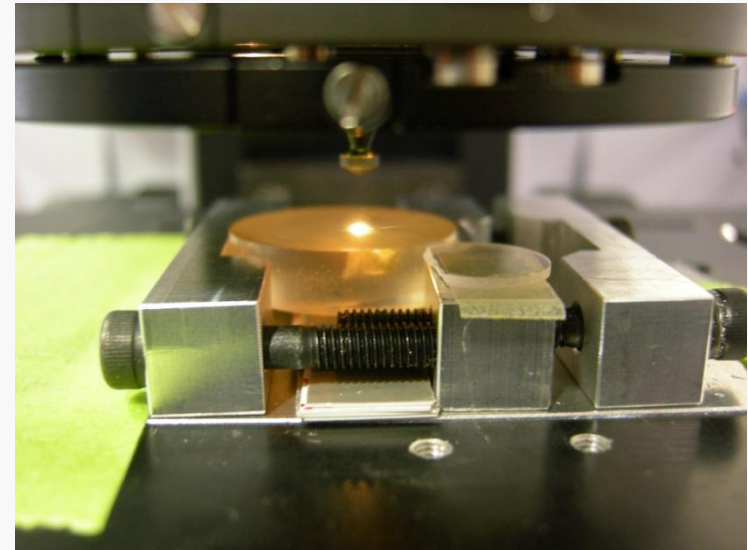
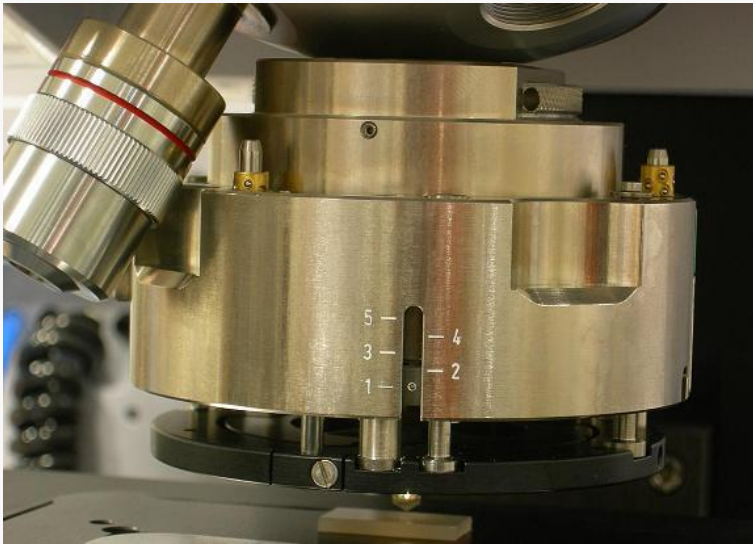


IR absorbance spectrum is generated when the IR beam reflects inside a crystal that is in contact with the sample

ATTENUATED TOTAL REFLECTION (ATR)



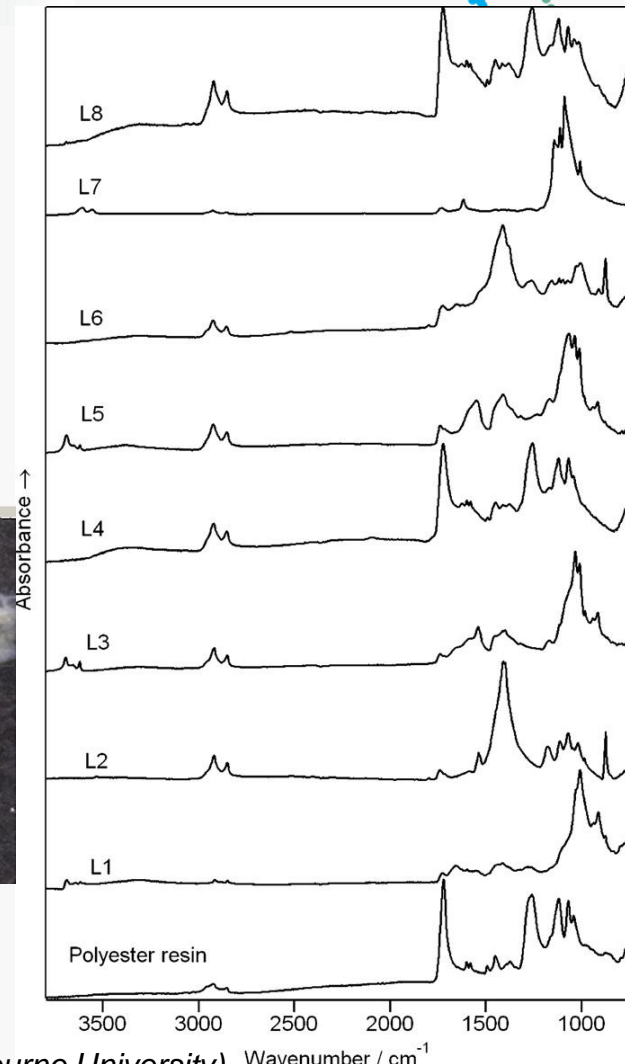
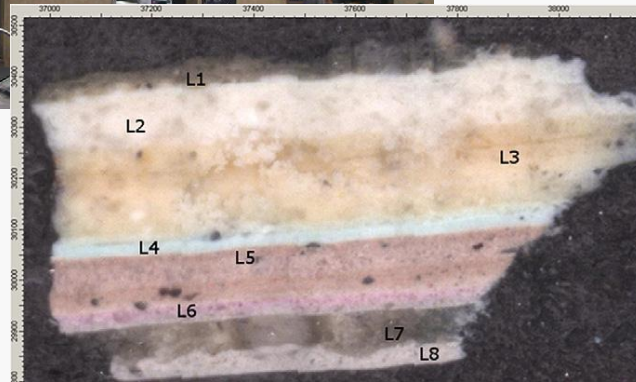
- Ge-crystal with ~100 microns crystal tip and variable pressure selection
- Contact between the crystal and the sample achieved by moving of the stage in z direction
- High spectral quality and spatial resolution



ANALYSIS OF PAINT LAYERS FROM A 19TH CENTURY BUILDING

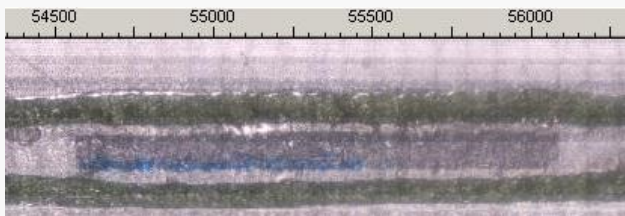
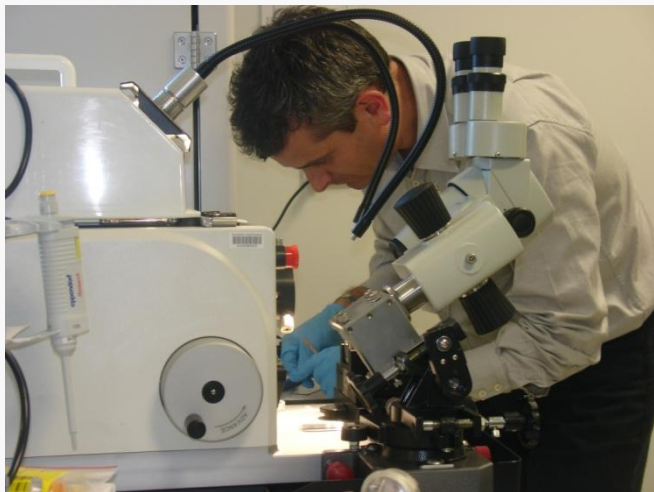


- cross-sections of paint chips from the Provincial Hotel in Fitzroy embedded in polymer.
- Information on pigment, binder and filler distribution.



Nicole Tse, Caroline Kyi, Robyn Sloggett, Stephen Best (CCMC and Melbourne University)

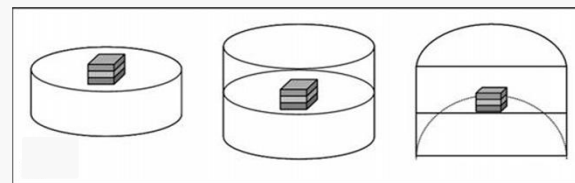
SAMPLE PREPARATION AVAILABLE AT THE AUSTRALIAN SYNCHROTRON



MICROTOMING FOR TRANSMISSION MEASUREMENTS



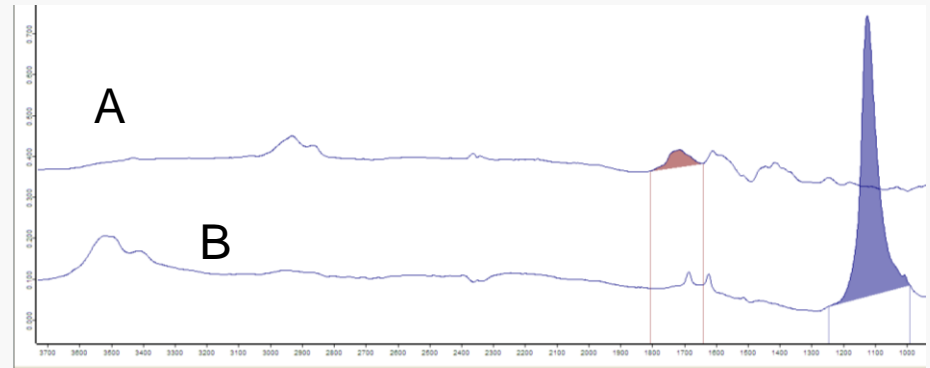
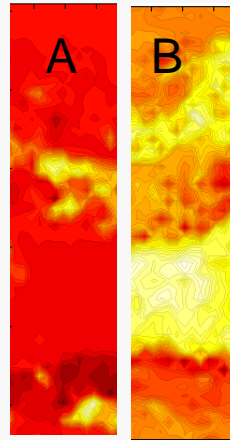
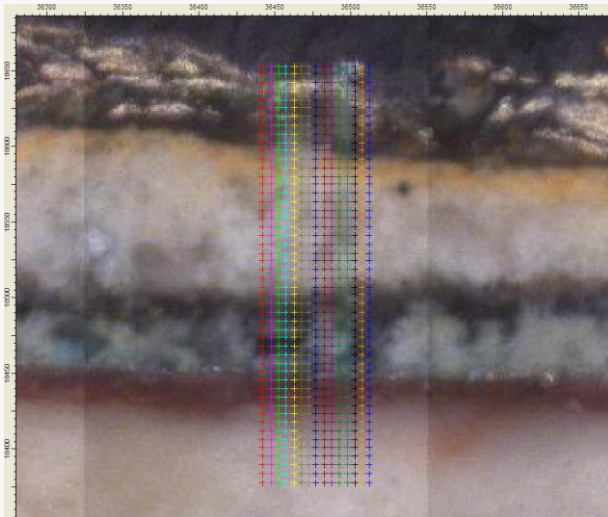
EMBEDDING THE PAINT SAMPLE IN RESIN AND POLISHING



EMBEDDING IN IR TRANSPARENT MATERIALS

DATA ANALYSIS

- Mapping cross sections embedded in resin



FTIR images showing the distribution of the integrated absorbance between certain wavenumbers

For analysis of large data sets can use statistical methods:

- Principal component analysis
- Cluster analysis

CONCLUSIONS

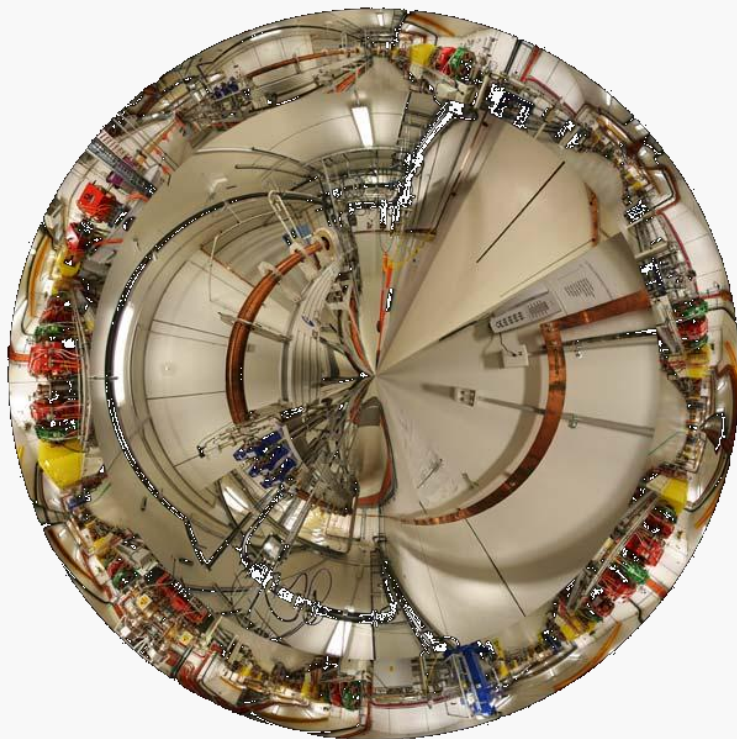
- Very useful tool for a range of materials and artefacts of cultural heritage interest.
- The Synchrotron brightness provides improved data quality and spatial resolution.
- Information on both organic and inorganic components (binders, varnishes, pigments and fillers) in a single analysis.
- Complementary to other techniques such as Raman spectroscopy, XRD and X-ray fluorescence.
- Sample preparation is still critical to obtaining the best results.

ACKNOWLEDGEMENTS



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- Melbourne University:
 - Stephen Best
- Australian War Memorial and Canberra University:
 - Alana Treasure, Dudley Creagh
- Queensland University of Technology, Brisbane :
 - Rosemary Goodall

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