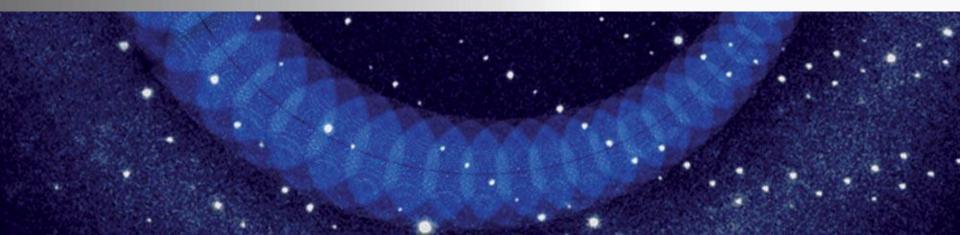




# SYNCHROTRON POWDER DIFFRACTION

**Kia Wallwork** Synchrotron Science for Cultural Heritage Materials, September 2010



# OUTLINE

#### **Powder Diffraction**

- Introduction & Applications
- Beamline Capabilities
- Data Quality



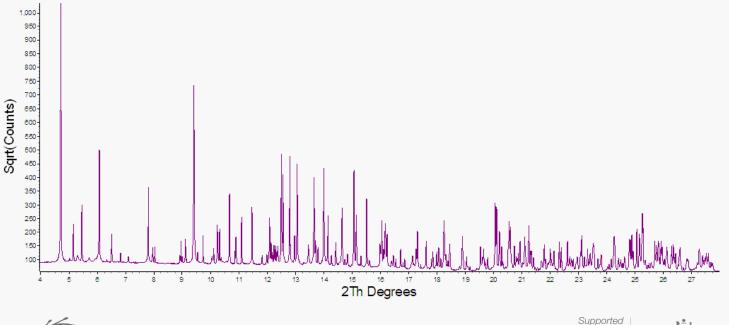
Supported by





# POWDER DIFFRACTION

- A technique for investigating atomic crystal structure
  - PD is not suitable for non-crystalline materials
- PD is not a spectroscopic technique
  - The peaks are not informative in isolation





by



# APPLICATIONS OF POWDER DIFFRACTION

- 1. Phase identification
- 2. Quantitative phase analysis
- 3. In situ examination of reaction processes

e.g. effect of heat treatment



Supported





# **TRADITIONAL X-RAY SOURCES**

Vital for thorough studies

#### Advantages

- Convenient to access
- Low cost
- Excellent for routine phase identification
- Quantitative analysis
- Some structure solution possible

#### Limitations

- Poor peak resolution
- Low intensity source
- Speed of data acquisition
- Limited sample ancillaries / stages



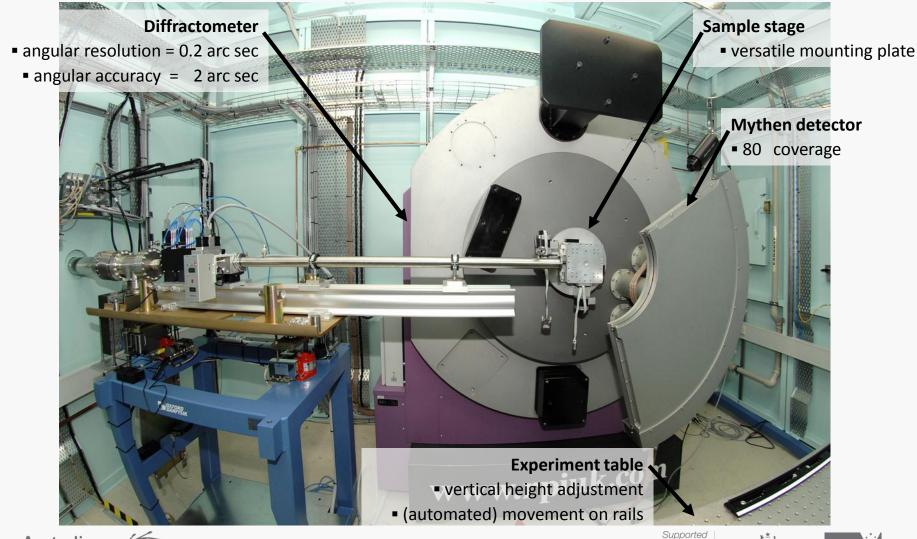


Supported





## POWDER DIFFRACTION BEAMLINE END STATION



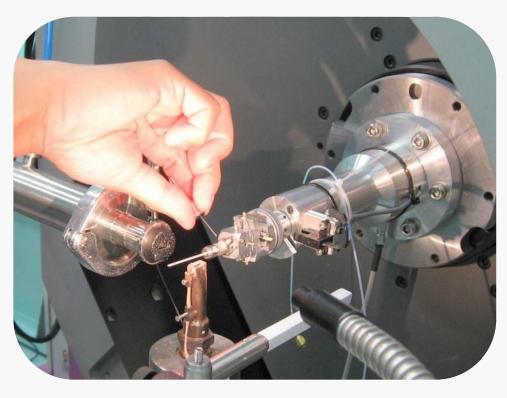
by

Australian Government

**AUSTRALIA** 



# SAMPLE ANCILLARIES – CAPILLARY SAMPLES





#### Temperature range = 80 - 1,173 K









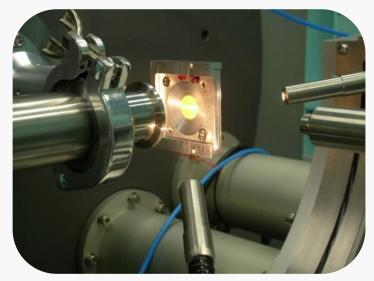


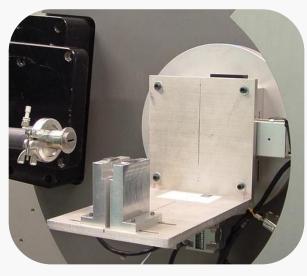
# SAMPLE STAGES – VARIOUS MOUNTING METHODS

















# MECCANO & STICKY TAPE





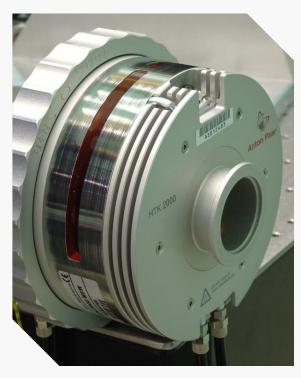






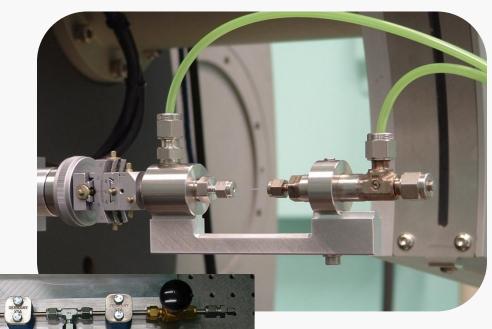
Australian Government

# SAMPLE ANCILLARIES – FURNACE & GAS FLOW



Furnace temperature range = 298 – 2,573 K







Supported by





Australian Government

# SEARCH / MATCH DATABASE

	🎕 Sleve+ - (Untitled)
Diffraction Pattern - f11_shell_p1_0000.xye	File Edit Matches Phases Lines Help
Edit Plots Window Help	Participanti de la companya de la
	Matches (3,196 of 291,440)
) 🖶 🍛 🗵	GOM PDF # QM Status Coords Compound Name Chemical Formula D1 D2 D3 D4
	7653 01-087-2096 S A ✓ Silicon Oxide Si O2 3.342960 4.254520 1.817630 2.456350 2.25
625,000	7646 01-086-2237 I A ✓ Silicon Oxide Si O2 3.342970 4.254780 1.817620 2.280930 1.54
600.000 -	7643 04-008-8228 P A ✓ Silicon Oxide Si O2 3.342810 4.254350 1.817550 1.541220 2.2
575,000	7637         01-085-0504         I         A         ✓         Silicon Oxide         Si O2         3.342940         4.254610         1.817610         1.541300         2.4           7637         01-085-0503         I         A         ✓         Silicon Oxide         Si O2         3.342940         4.254610         1.817610         1.541300         2.4           7637         01-085-1053         I         A         ✓         Silicon Oxide         Si O2         3.342940         4.254610         1.817610         1.541300         2.4
550,000 -	7637 01-065-1051 H X → 3illicon Oxide Si O2 3.342940 42.59610 1.617540 1.541330 2.4 7635 04-065-6050 P A → Sillicon Oxide Si O2 3.343000 42.54700 1.541330 2.4
	7633 04-005-4494 B A ✓ Silicon Oxide Si O2 3.342680 4.254250 1.817470 1.541180 2.4
525,000 -	
500,000	Matches Filter Filter Description
475,000 -	
450,000	Select
425,000	Experiment
400.000 -	Search Line(s): 3.34332 ▼ Å D1 Range: 3.311 - 3.376 Å Rotation: All Ex.d(Å) ▼ Ex.I P1.d(Å) P1.I
375,000	
	Preferences 4 03854 3
350,000 -	Search Method: Hanawalt Wavelength: ko1 0.88397Å
325,000	Search Window:         0.15         •         Match Window:         0.15         •         1         2.45618         6         2.456350         6
300,000 - 275,000 -	Lowest Allowable GOM: 2000
275,000 -	Phases (1) 2.23641 3 2.236220 3 2.12717 4 2.127260 4
250,000 -	# ▲         Accepted         PDF #         Compound Name         Int. Ratio         Int. %         I/Ic         Time         IIII 100         IIII 100         IIIII 100         IIIII 100         IIIIII 100         IIIIIIII 100         IIIIIIIIIIIIIIIIIIIIIIIIIIIIIIIIIIII
225,000	Composition Andre Antroposition Antropositio Antroposition Antroposition Antroposition Antroposition Antroposit
200.000 -	1.67121 3 1.671480 3
	1.54094 8 1.541280 6 1.38180 5 1.381890 3
175,000	
150,000 -	Processing Pattern GOM's192 of 3,196 Cancel
125,000	Processing Integral Indexes3,196 results Cancel
100.000	
75,000	
50,000 -	
25,000	
-25,000	·····································
-50,000 -	
0 5 10	15 20 25 30 35 40 45 50 55 60 65 70 75 80 85 20



.



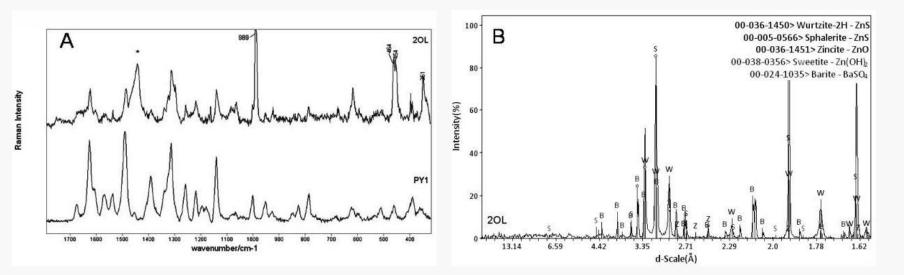


Combined X-ray Diffraction and Raman Identification of Synthetic Organic Pigments in Works of Art: From Powder Samples to Artists' Paints

L. B. Brostoff, S. A. Centeno, P. Ropret, P. Bythrow, and F. Pottier, Anal. Chem. 2009, 81, 6096–6106

XRD

#### Raman



**Figure 4.** (A) Raman spectra of the oil bound sample 2OL (Lukas (Studio) Brillantgelb hell, top) and of a PY 1 reference sample (SunChemicals, bottom). Main bands are due to the extenders BaSO<sub>4</sub> (ca. 989 cm<sup>-1</sup>, shown out of range, 464 and 454 cm<sup>-1</sup>) and ZnS (ca. 351 cm<sup>-1</sup>) indicated. The band at ca. 1440 cm<sup>-1</sup>, marked by an asterisk, may arise from CH<sub>2</sub> deformations of the oil binder;  $\lambda_0 = 785$  nm. (B)  $\mu$ XRD pattern detail (Cr K $\alpha$  radiation) of paint sample 2OL, after background subtraction, with ICDD pattern matches shown in vertical bars (barite peaks marked with "B"; zincite peaks marked with "Z"; sweetite peaks marked with "S"; wurtzite peaks marked with "W"; sphalerite peaks marked with "S").

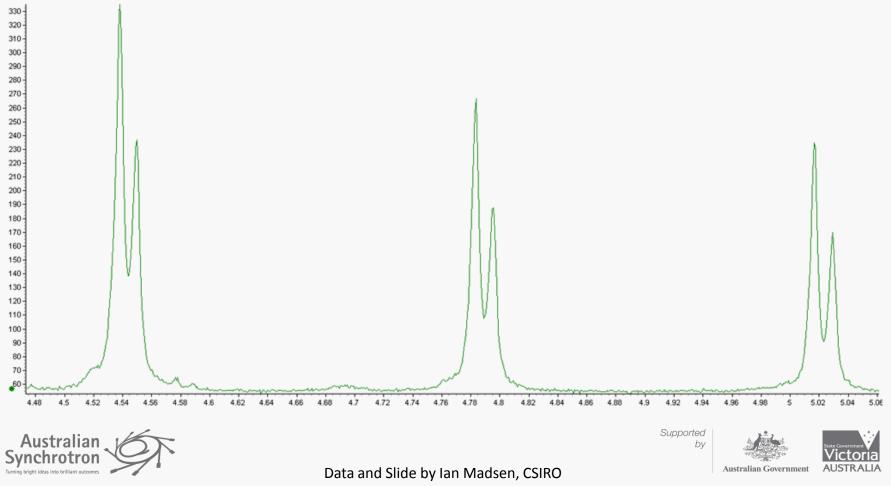




## DATA QUALITY

#### Laboratory XRD Instrument

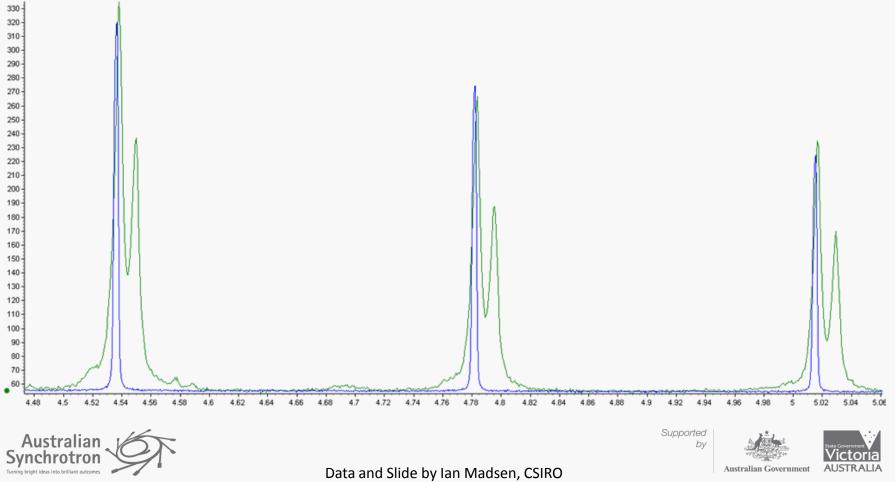
- Cu Kα<sub>1,2</sub>
- Stationary sample divergence in beam ensures good 'powder average'



## DATA QUALITY

#### Synchrotron PD data overlaying laboratory data

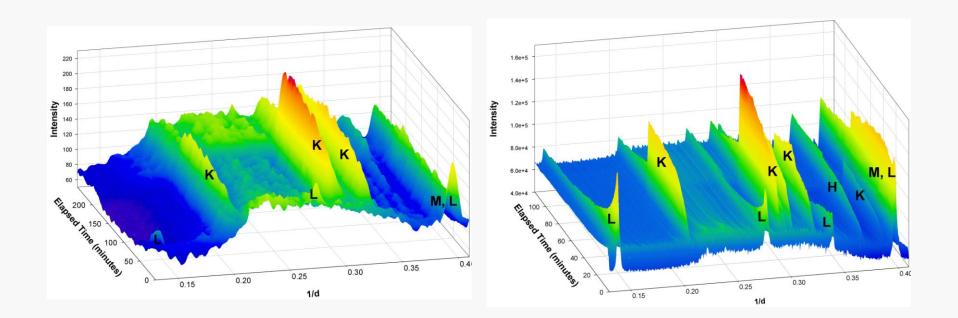
- 0.3mm capillary
- Spinning sample rotating sample ensures good 'powder average'



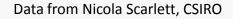
# SIGNAL TO NOISE

### Laboratory XRD

### Synchrotron XRD







Supported by





# TAKE-HOME MESSAGE

Synchrotron powder diffraction

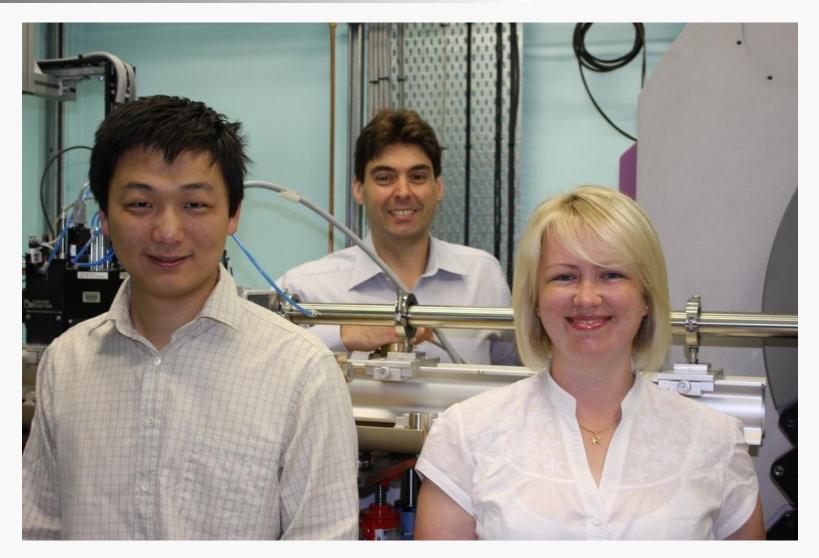
- Important tool for materials characterisation
- Non-destructive
- Flexible and tailored sample mounting options
- High resolution data
- High intensity X-rays
- Tunable, high energy X-rays







# THANK YOU





Supported by





Australian Government