University of Glasgow

CoSE Analytical Suite

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Analytical Services Manager

WORLD CHANGING GLASGOW

CoSE Analytical Suite

Establish cross-College (and beyond) analytical suite

- providing core characterisation for advanced materials
- expanding our experimental capabilities and upgrading existing
- EPSRC core equipment award (2020) used to kickstart facility
- grow diverse College wide user base
- encourage cross-College interactions
- Develop facility management

CoSE Analytical Suite

Phase 1 2020/21

- X-ray diffraction (powder, thin films) XRD
- Thermal Analysis (TGA & DSC)
- ICP-OES
- Raman Spectrometer
- CD spectrometer
- Nepholometer

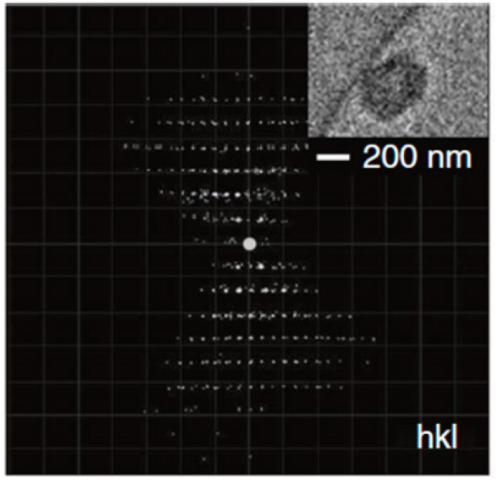
Phase 2 2023/4

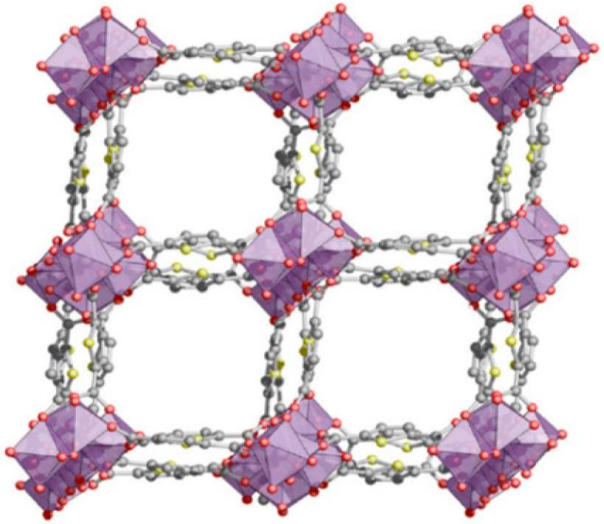
- MicroCT – X-ray computed tomography
- X-ray photoelectron spectroscopy (XPS)
- Electron diffractometer (ED)

https://www.gla.ac.uk/colleges/scienceengineering/staff/researchsupport/collegeanalyticalsuite/ scieng-analytical-suite@glasgow.ac.uk

3D Electron Diffraction

- Determines crystal structures of nanomaterials by their interaction with electrons
- Precise determination of atomic structure in crystalline samples
- Particles as small as 100 nm can be analysed
- Compare to single crystal X-ray diffraction which requires 10 micron crystals
- Samples run in a vacuum – must be solid-state and reasonably robust
- PXRD and SEM pre-screening (crystallinity and particle size)





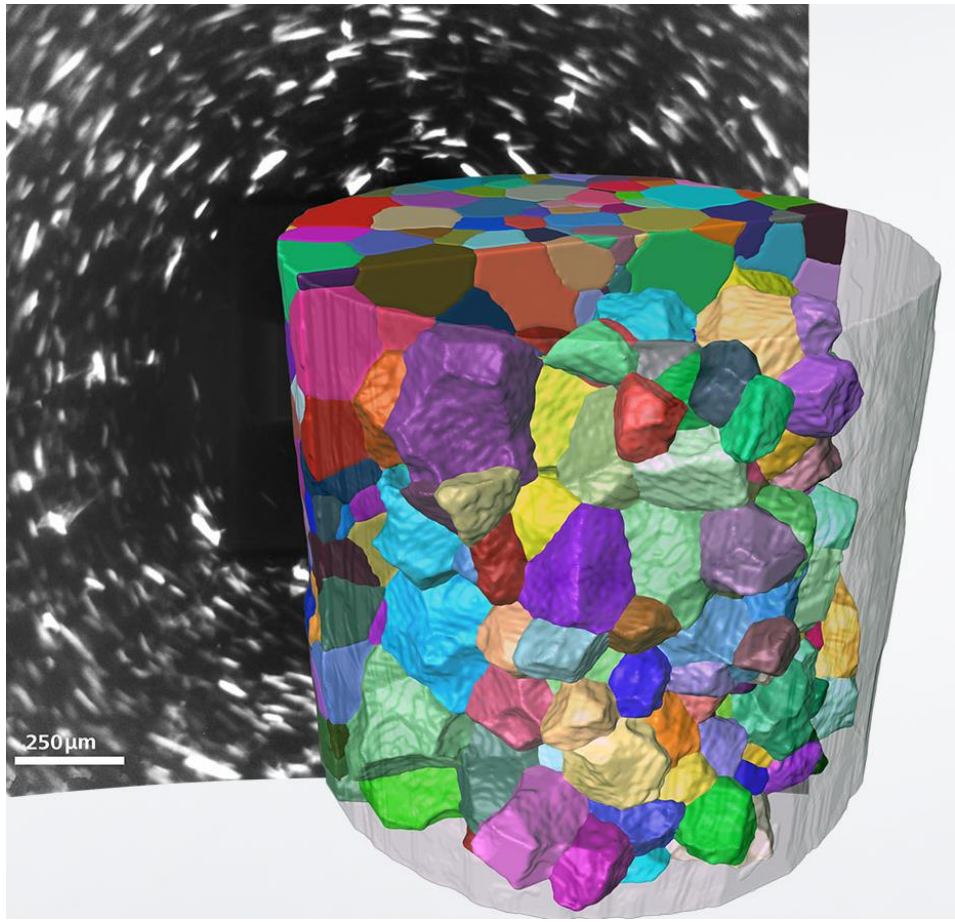
Our Electron Diffractometer

- Rigaku XtaLAB Synergy ED – delivery early 2024 – 3rd system in UK
- Requirements of the system:
  - Sample size: 100 nm
  - Variable temperature measurements
  - Cryo-loading stage (c.f. cryo-EM) for fragile or solvated samples
  - Automation of analysis of multiple samples across a single grid
  - Remote access to drive the diffractometer
  - Ease of use for existing X-ray crystallographers by using existing analysis software, simple user friendly set up

EPSRC Strategic Equipment Award: EP/X030083/1 (PI Prof Ross Forgan)

3D X-Ray Tomography

- Looks inside materials to determine their structures
- You can create cross-sections and virtual 3D models of your materials
- Non-destructive
- Sub-micron resolution
- Applicable to all sorts of materials, from biological samples through to electronic components, polymers and inorganic materials



Our Tomography Instrument

- Installed July 2023
- Zeiss Xradia 620 instrument with:
  - 500 nm resolution at distances from source-to-sample of 5 cm or more, allowing items the size of a head to be imaged in exquisite detail
  - Max sample mass = 25 kg
  - Inert atmosphere capability
  - In situ electrochemical measurements (e.g. for batteries)
  - Possibility for in situ pressure and flow measurements
  - Variable temperature
  - Full software for 3D reconstructions

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EPSRC Strategic Equipment Award: EP/W02134X/1 (PI Prof Mark Symes)

XPS (X-Ray Photoelectron Spectroscopy)

- Looks at the surfaces of samples using the photoelectric effect
- XPS can identify the elements on a surface, as well as their chemical state, and the overall electronic structure and density of the electronic states in the material.
- So you can see what elements are on a surface and also what they are bound to.
- Samples are held under high vacuum, and so this technique works best for solid state samples.

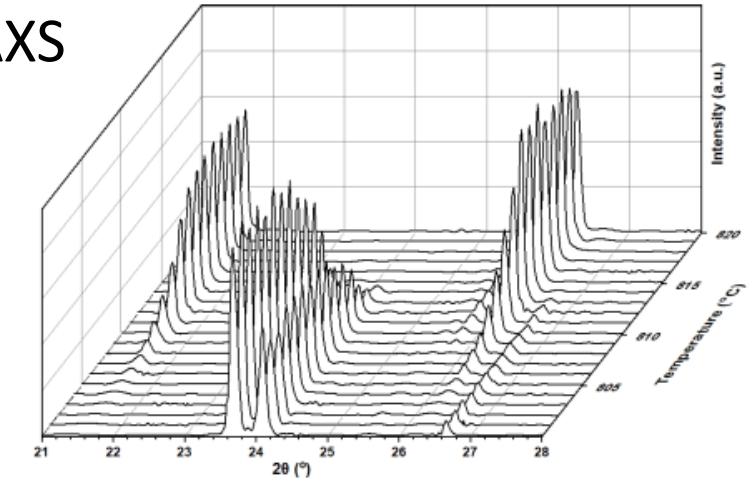
Our XPS Instrument

- Installed and initial use Nov 23
- Is a Kratos Axis Supra+ instrument with:
  - Ultraviolet photoelectron spectroscopy (UPS) – to determine materials' work functions
  - Reflected electron energy loss spectroscopy (REELS) - to determine materials' surface valence and conduction band electronic properties
  - Inert atmosphere holders and transporters
  - In situ electrochemical measurements (e.g. for batteries)
  - Variable temperature
  - A gas cluster ion source for depth profiling of organic and inorganic multilayer thin films

EPSRC Strategic Equipment Award: EP/W02134X/1 (PI Prof Mark Symes)

XRD – X-ray Diffraction/Scattering

- Measure x-ray diffraction and scattering from range of sample types – powders, thin films, non-crystalline samples
- You can use for phase id (materials, match to crystal structure, minerals), crystallography – structure determination
- Measure nanoscale structure of solids/liquids through SAXS (small angle x-ray scattering)
- Study thin films – phase, thickness of layers (Grazing incidence XRD, XRR X-ray reflectometry)
- Reciprocal Space Mapping of epitaxial samples
- Monitor changes with in-situ non-ambient studies



Stacked PXRD Patterns of $BaCO_3$ collected at 800 – 820°C

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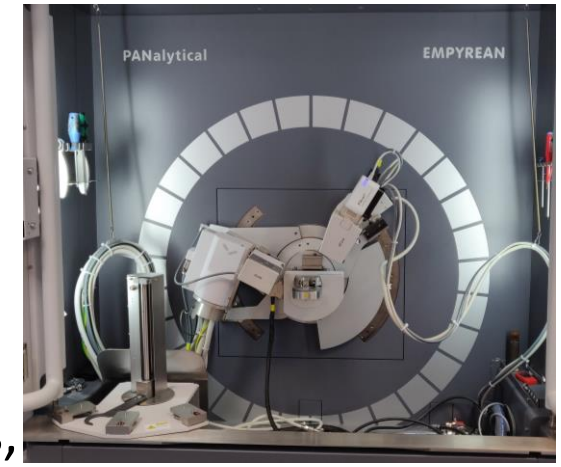
Our XRD Instrument



Is a Malvern Panalytical Empyrean multi-purpose platform installed in Dec 2021 in the Joseph Black Building

- Cu sealed tube X-ray source, multicore iCore/dCore motorised optics, hybrid monochromator, focussing mirror
- PIXcel^{3D} detector

Very versatile with many configurations including:

- Reflection and transmission geometry with 45-position sample changer, capillary stage
- 3-axis cradle
- Grazing Incidence XRD , X-ray reflectometry (thin films)
- Anton-Paar XRK900 non ambient chamber 25 <T <900 °C under gas flow
- SAXS

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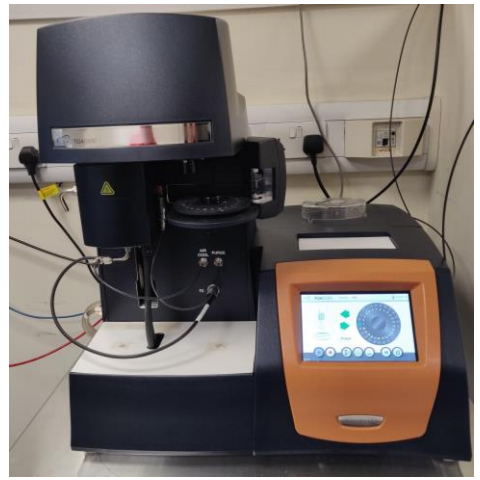
TGA & DSC – thermogravimetric analysis & differential scanning calorimetry

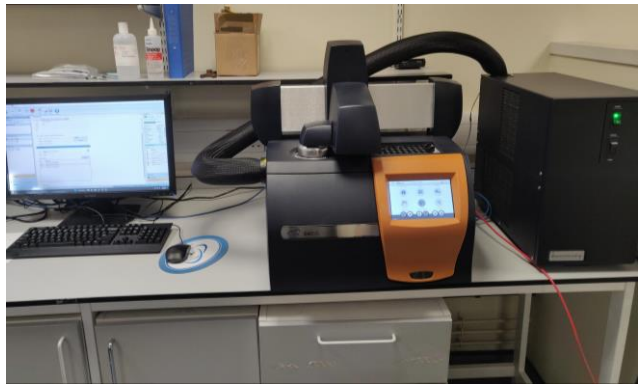
- techniques to monitor response of a sample to temperature characterise thermal behaviour of a range of materials
  - TGA - phase changes, adsorption/desorption processes, thermal decomposition
  - DSC – information on energetics of temperature dependent processes (including those with no changes in mass)
- Information that can be attained:
  - Melting point, crystallinity, curing and cross linking, heat capacity, purity, thermal stability, oxidative stability

Our TGA and DSC instruments



TA Instruments

- TGA 5500 – temperature range: ambient to 1200°C
- DSC 25 with RCS90 refrigerated cooling system; temperature range: -90 – 725 °C
- SDT Q600
  - temperature range: ambient to 1200°C
  - gas atmospheres available: Argon, O_2 mix, H_2 mix
  - EcoCat 200D portable mass spectrometer system



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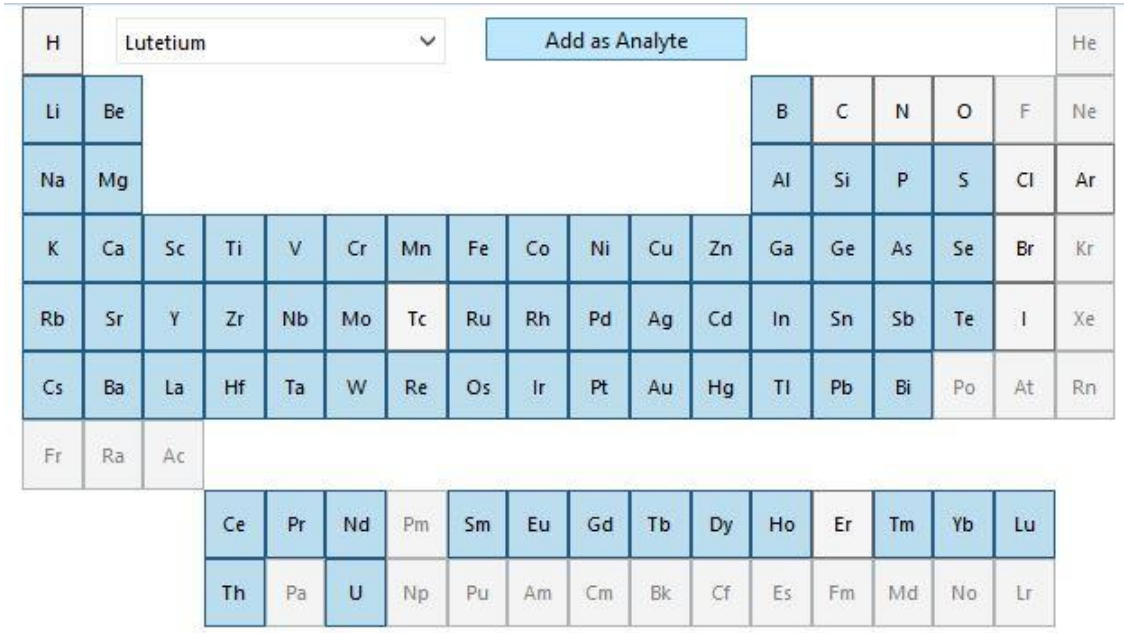
ICP-OES –

Inductively Coupled Plasma Optical Emission Spectroscopy

- Elemental analysis technique based on sample decomposition in high temperature plasma and measurement of emission of excited atoms/ions at characteristic wavelengths
- The following 70 elements can be detected using the ICP-OES:
- Versatile technique for determining elemental composition of diverse materials

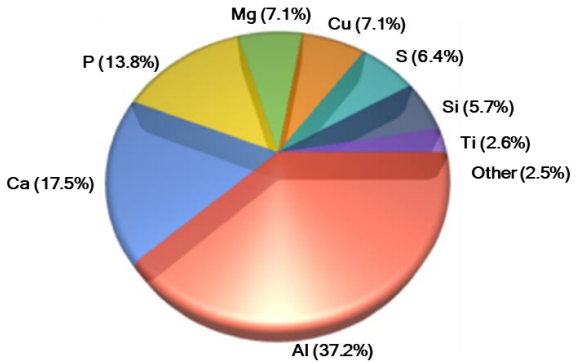


Our ICP-OES instrument

Inductively Coupled Plasma Optical Emission Spectroscopy

Agilent 5900 SVDV ICP-OES

- Quantitative analysis of all 70 elements with detection limits down to a few ppb
- Multiple elements can be quantified simultaneously
- High-throughput valve system allows samples to be analysed in less than a minute, so dozens can be measured in a single run
- Semi-quantitative screening tests can identify elemental composition of unknown samples without the need for sample-specific calibrations



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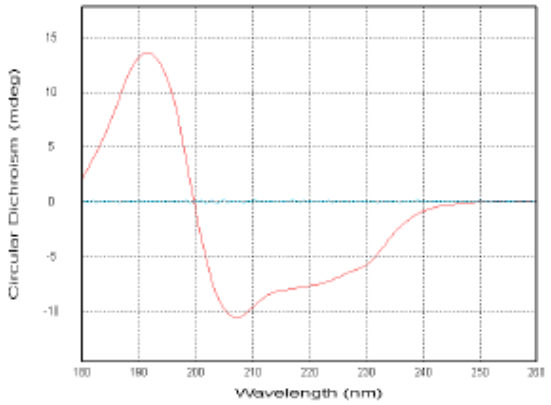
Circular Dichroism Spectrometer

Circular Dichroism Spectrometer

- spectroscopic technique used to characterise chiral materials
- Chiroptical response of nanomaterials
- Conformation analysis of peptides and peptidomimetics



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Our Circular Dichroism Spectrometer

Applied PhotoPhysics Chirascan VX

- Wavelength range from 160 nm up to 950 nm. Typically, owing to variation of CD amplitude with wavelength, narrower ranges are used such as far-UV (ca. 160-250nm) for protein secondary structure and near-UV (250-350nm) for tertiary structure.
- Peltier temperature controller to maintain sample/sample chamber at constant temperature.
- Can also perform continuous or stepped temperature ramps up to 100°C, with single wavelength or multi-wavelength scans to determine thermodynamic properties.
- Range of measurement cells – 10 mm stoppered cuvette and 0.5 mm stoppered cuvette with 10 mm adaptor are currently available. Cuvettes down to 0.01 mm path length can be fitted – an adaptor for thinner cuvettes is already available but the cuvettes themselves must be ordered.

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Raman Spectroscopy

Raman Spectroscopy is a non-destructive chemical analysis technique which provides detailed information about chemical structure, phase and polymorphy, crystallinity and molecular interactions. It is based upon the interaction of light with the chemical bonds within a material.

Typical areas where Raman is used today

- Art and archaeology – characterisation of pigments, ceramics and gemstones
- Carbon materials – structure and purity of nanotubes, defect/disorder characterisation
- Chemistry – structure, purity, and reaction monitoring
- Geology – mineral identification and distribution, fluid inclusions and phase transitions
- Life sciences – single cells and tissue, drug interactions, disease diagnosis
- Pharmaceutics – content uniformity and component distribution
- Semiconductors – purity, alloy composition, intrinsic stress/strain

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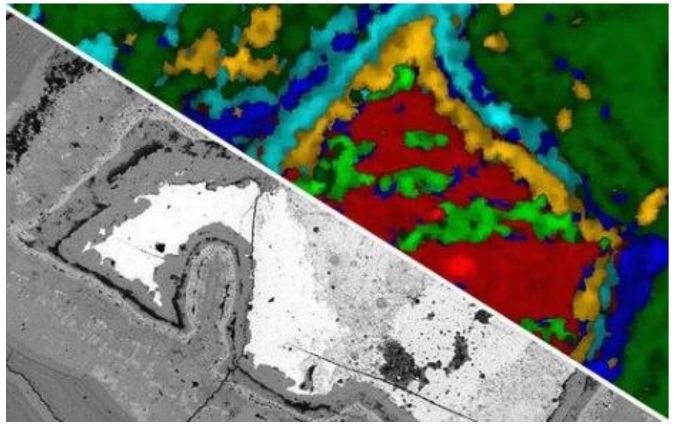
Our Raman Spectrometer



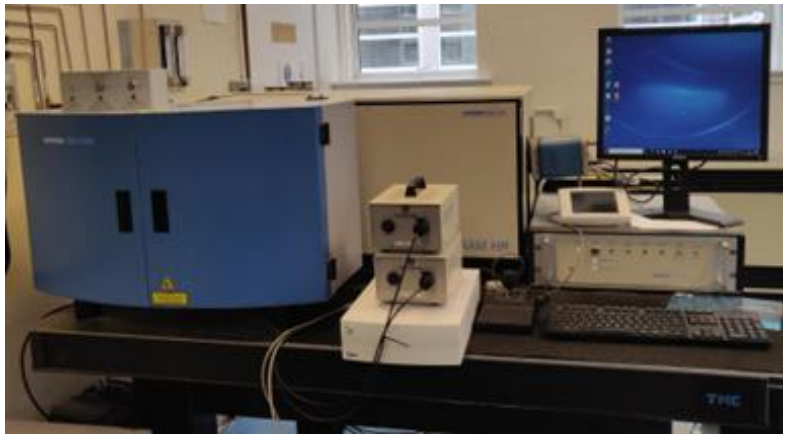
Horiba Jobin Yvon LabRAM HR system with the following lasers

- Ventus 532 laser system, 250mW, 532nm
- Helium Cadmium IK3201R-F, 20mW, 325nm

It uses a Synapse CCD detection system, and has the following objectives:

- 40x for UV; 10x, 50x and 100x for visible

- A Linkam TS 1000 High Temperature Stage is also available.



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Nepholometer

Nepholometer

- Analytical instrument to determine compound solubility in liquid samples by measuring forward scattered light
- Drug solubility determination, flocculation assays, bacterial and fungal growth kinetics
- We have a BMGLabtech Nephelostar Plus

CoSE Analytical Suite

- available to users across CoSE and the University, and to external use both academic and commercial
- generally, open access to trained users via online booking systems or as a service for small number of samples/infrequent use
- charging models in place for all the equipment and scope for initial measurements as a feasibility check or to inform a funding proposal

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More Information

- All instruments will be available for general use across the University and for external users via the College Analytical Suite. Users will be able to run their own samples after training.
- Access charges for academic users will be in-line with those advertised by the EPSRC national facilities for XPS and X-ray tomography. **Electron diffraction will be free for the first three years for academic users.**
- XPS: https://www.kratos.com/products/axis-supra-xps-surface-analysis-instrument
- Tomography: https://www.zeiss.com/microscopy/en/products/x-ray-microscopy/xradia-versa.html#Versa_620
- ED: https://www.rigaku.com/products/crystallography/synergy-ed
- **Contacts.** General enquiries: scieng-analytical-suite@glasgow.ac.uk or Claire Wilson (claire.wilson.2@glasgow.ac.uk)