EURO-CARES

A PLAN FOR EUROPEAN CURATION OF RETURNED EXTRATERRESTRIAL MATERIALS

WORK PACKAGE 4

INSTRUMENTATION

(DELIVERABLE D4.2)

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1- Objectives

The overall objective of work package 4 is to establish the most appropriate chain of analyses to perform within the Curation Facility whilst maximising preservation of the samples and minimising contamination for efficient distribution of samples to the scientific community. These analyses will require specific, and dedicated instruments in order to achieve a range of goals such as sample characterisation, contamination control and knowledge, sample selection and preparation. As part of this effort the instrumentation required within the Curation Facility will be identified. This report provides a preliminary report of the instrumentation that has been identified, as well as some of the performance and service provision requirements. Instrumentation required for Planetary Protection purposes (i.e. bio-burden, bio-hazard assessment) is currently within the remit of WP2. Amalgamation of the instrumentation required as part of WP4 and WP2 will be made later in the project.

It needs to be stressed that this is a preliminary report, drawn primarily from literature, experience of the authors and initial discussions within the EURO-CARES project. The report is generated now in order to provide some initial input into other WPs, including; WP2 to help identify overlap between the requirements of this WP and the instrumentation required for Planetary Protection activities; WP3 to help constrain the design requirements for the curation Facility; and WP5 to help define the analogues required for both sample handling/processing and contamination control.

The output from this task is also required for WP4 in order to help identify suitable manufacturers for discussion about optimum solutions and identification of any gaps between instrument requirements and capability so that future technology developments can be specified.

2- Summary of Requirements

Drawing upon Deliverable 1.4, the instrumentation is required to undertake a series of activities. These include:

1) *Characterisation of the samples*. This covers a wide range of measurements; from photo-documentation of the samples primarily for identification purposes and detailed records of the samples sent out, to preliminary determination of the structure, mineralogy and organic inventory of the samples, while detailed measurements of this type would be expected to be an activity undertaken by the scientific community on allocated samples. All such activities in the curation facility should be conducted with little, or no, impact on the physical and chemical nature of the sample.
2) *Sample selection and quality control on sample preparation*. Identification and verification of the most appropriate samples to meet the requirements of approved sample requests, and if specific sample preparation is required (e.g. polished sections) that the samples have been prepared properly.
3) *Monitoring of clean room operations*. This will include analysing witness plates and test samples in order to verify that samples are not exposed to unacceptable levels of contamination and that cleaning and handling procedures are meeting specification.

3- Types of Instrumentation

Each instrument may be involved in several different types of activities or measurements, these are identified in the information for each instrument provided in Section 4 - Instrument Descriptions. While it would be intuitive to organise the instruments by activity, this would result in duplication or cross-referencing, and therefore an alternative approach is adopted here. The wide range of instruments required in an advanced Curation Facility employ many different approaches, measuring a wide range of physical, spectral and chemical properties. However, of prime importance is how the instruments interact with the sample and how that interaction may modify or contaminate the sample. Therefore, the instrumentation has been organised by the nature of the measurement. Summarised from Deliverable 1.4, these categories are as follows:

1) *Optical methods for documentation.* Such methods are strictly non-destructive but may still require destructive sample preparation, such as polished thin sections. These tools are necessary primarily for the documentation and very preliminary characterisation of the samples. For instance, it will be necessary to characterise the size, shape, texture, colour, albedo etc. It is anticipated that optical documentation of samples at all stages of processing will be a frequent requirement, resulting in a large volume of data, and therefore high levels of automation, including data handling, will be an important aspect of these instruments. Examples: *macro imaging; optical microscopes (including large depth of field), scanning near-field optical microscopy, 3D optical shape profiling*

2) *Methods for characterisation of physical properties*. Required to provide additional information relating to the physical nature of the samples. These techniques are generally non-destructive, usually requiring no sample preparation although some subtle modification of the sample may be possible. Among the properties analyzed by these techniques are the mass of grains and fragments, density, grain density and porosity and internal structure of fragments at different scales. Examples*: balances, x-ray CT.*

3) *Spectroscopic methods.* These techniques provide the characterisation of the mineralogy and chemical composition/nature of the samples with minimal sample handling, and sample preparation. They are minimally damaging but power of the incident radiation must be controlled (e.g. Raman spectroscopy) and some sample preparation can be required. Some measurements may be performed on samples within sample canisters, offering great control on contamination and ready application to bio-hazardous samples, although potentially impacting on data quality and sample canister design. Examples*: Fourier Transform Infra Red (FTIR) spectroscopy, Raman spectroscopy, VIS-NIR bidirectional spectroscopy, x-ray diffractometer.*

4) *Scanning and electron probe methods*. These types of instruments provide high-resolution morphological information (beyond optical) as well as detailed chemical and structural information. These techniques can be minimally damaging, with the electron beam typically affecting the outer few μm of the sample. Sample contamination can be a major issue, with the potential for electron beam induced deposition and exposure to potentially reactive gases or coatings to obviate charge built. While such instruments might generally not be used on the most pristine samples, they are necessary for advanced sample preparation and for the assessment of witness plates used for monitoring particulate matter to identify sources of particles. Examples*: Scanning Electron Microscopy (SEM) and associated interactions/detections such as EDX (energy dispersive x-ray spectroscopy) and possibly CL (cathodoluminescence), or EBSD (electron backscattered diffraction). Transmission Electron Microscopy (TEM) and associated techniques such as Focused Ion Beam (FIB) for sample preparation are highly specific and normally would only be considered for specific sample types.*

5) *Chemical methods and other destructive techniques* This category groups all instrumentation required to undertake chemical analyses of the samples, either as part of the sample characterisation program or for more advanced/detailed measurements identified as needing to be performed within the ESCF. However, their more useful activities will be in the characterisation and monitoring of contamination of the environment and procedures within the Curation Facility. Examples: *Time of flight - Secondary ion mass spectrometry (TOF-SIMS), Gas chromatography and liquid chromatography - mass spectrometry (GC-MS and LC-MS), Inductively coupled plasma MS (ICPMS), Elemental Analyser (EA).*

The following sections describes each of the instruments identified as required for a sample curation facility handling primarily rocky samples returned from a COSPAR unrestricted category V target/body. Additional requirements for restricted missions are discussed in D2.1 and D2.2. A major emphasis has been on identifying the minimum number of instruments required to provide the necessary documentation and initial characterisation, sufficient to provide enough information to the scientific community to request samples for scientific investigation and for the curation team to select

the most appropriate samples to meet each request. This approach minimises cost of the instruments required for the curation facility, as well as the cost of the laboratory space to accommodate the instruments, operating costs (i.e. consumables, service costs, etc), staff support costs (which can be very expensive over the duration of a curation facility) and the necessary infrastructure to support the staff. This approach also minimises sample handling and interaction, thereby minimising possibility for contamination and sample disruption/modification.

4- Instrument Descriptions

4.1 Optical Methods

Most samples that will likely be returned to a future curation facility will be complex, and comprised of grains ranging in size from cm to sub-micron. A range of optical microscopes will be required to characterise and document the different types or components.

LOW MAGNIFICATION OPTICAL MICROSCOPE	
Principle measurements	Macro examination of samples Photo documentation of irregular shaped samples at multiple scales (from mm to 10 micron resolution)
Instrument specifications	Magnification from x5 to x100 Large depth of field (including camera) Working distance ≥20mm (to allow observation in canister) Record high resolution full colour images XYZ stage control (10 micron repeatability) Remote operation
Sample requirements	Sample can remain in sample canister
Effect on sample	None, but careful handling with dedicated material of the sample and/or canister is crucial.
Links	Sample orientation shared with spectral imaging, 3D imaging
Data output	Multiple MB images per sample
Service requirements	Lab electric supply
Examples	Microscopes - Zeiss/Nikon/Leica/etc (e.g. Leica M205A) Same manufacturers can provide high quality image cameras.
Other info	Camera system required – conventional cameras provide narrow depth of field, but automation can stack up multiple exposures at different height or focal adjustment to provide continuous image. Better solution may be to 3D light field cameras that can provide high depth of field/3D images – Raytrix (https://www.raytrix.de/produkte/#r12microseries). Technology development to integrate this data source into a virtual microscope type system for hand specimen reconstruction including accurate shape model of each sample –requires integration of rotating stage (or mount camera system on system to move around sample – requires assessment of best approach to minimise contamination). Multiple low magnification stereo microscopes may be required within the facility for a range of processes and at different locations within the facility.

HIGH MAGNIFICATION OPTICAL MICROSCOPE - MATERIALS	
Principle measurements	Micro examination of samples Photo documentation of samples at multiple scales (from 10 micron to sub-micron resolution)
Instrument specifications	Magnification from x15 to x1000 Large depth of field (including camera) Working distance ≥20mm (to allow observation in canister – at least at low magnifications)) Record high resolution full colour images XYZ stage control (sub-micron repeatability) Transmitted and brightfield/darkfield illumination Remote operation
Sample requirements	Sample can remain in sample canister
Effect on sample	None
Links	Sample orientation shared with spectral imaging, 3D imaging
Data output	Multiple MB images per sample
Service requirements	Lab electric supply
Examples	Microscopes - Zeiss/Nikon/Leica/etc (e.g. Leica DM6 M) Same manufacturers can provide high quality image cameras.
Other info	3D light field cameras can provide high depth of field/3D images – Raytrix (https://www.raytrix.de/produkte/#r12microseries). Technology development to integrate this data source into a virtual microscope type system.

HIGH MAGNIFICATION OPTICAL MICROSCOPE - PETROGRAPHIC	
Principle measurements	Micro examination of samples Photo documentation of samples at multiple scales (from 10 micron to sub-micron resolution)
Instrument specifications	Magnification from x15 to x1000 Large depth of field (including camera) Working distance ≥20mm (to allow observation in canister – at least at low magnifications)) Record high resolution full colour images Rotation & Z stage control (sub-micron repeatability) Transmitted and brightfield polarised illumination Remote operation
Sample requirements	Sample can remain in sample canister
Effect on sample	None
Links	Sample orientation shared with spectral imaging, 3D imaging
Data output	Multiple MB images per sample
Service requirements	Lab electric supply
Examples	Microscopes - Zeiss/Nikon/Leica/etc (e.g. Leica DM4 P) Same manufacturers can provide high quality image cameras.
Other info	3D light field cameras can provide high depth of field/3D images – Raytrix (https://www.raytrix.de/produkte/#r12microseries). Technology development to integrate this data source into a virtual microscope type system.

Documentation of shapes can be achieved by recording the 3D shape of each particle. This information not only provides a detailed measure of the volume (that can therefore provide macro density) but will also allow re-construction of the spatial relationships of sub-fragments after sub-sampling or un-planned fragmentation during sample storage or handling. Using optical techniques should permit such measurements in canister and/or ultra-clean conditions.

3D OPTICAL SHAPE PROFILER	
Principle measurements	Sample roughness is reconstructed from interference fringes created from optical path difference between light reflected from the studied surface and from a reference surface, respectively
Instrument specifications	Surface topography repeatability from 0.02 to 3.5 nm Spot size from sub-micron to few micron Scan speed from 10 µm/s to 100 µm/s Maximum step height <20 mm (<95 µm for portable profilers) FOV between 9 and 16 mm
Sample requirements	Sample can remain in sample canister
Effect on sample	None (non-contact technique)
Links	Spectral and optical imaging
Data output	3D topography map (1024x1024 or 2048x2048)
Service requirements	Lab electric supply
Examples	ZYGO profilers (http://www.zygo.com/?/met/profilers/) Taylor-Obcon profilers (http://www.taylor-hobson.com/products/23/109.html)
Other info	This instrument may not be required if x-ray tomography is used, although the acquisition times for this can be orders of magnitude longer with current technology and therefore unlikely to be used on all samples/sub-samples. Combining 3D shape with optical images can be achieved readily (e.g. macro-imagine virtual microscope). Further enhancement of the 3D virtual microscope technology may be possible incorporating 3D light field cameras (e.g. https://www.raytrix.de/produkte/#r12microseries) for enhanced image quality and improved data acquisition time – some technology development required to integrate this data source into a virtual microscope type system.

SCANNING NEAR-FIELD OPTICAL MICROSCOPE (SNOM)	
Principle measurements	A local scatterer is placed in resonant oscillation close to the sample surface, allowing to reconstruct its topography and to retrieve the sample reflectance/transmittance image at one or more wavelengths
Instrument specifications	Spatial resolution better than the diffraction limit (λ/2), down to λ/60 Numerical aperture of 1.3-1.4 Images of a few um-sized regions Acquisition time (for a 100x100 image): few minutes for topography, imaging acquisition can vary from few to 15-20 minutes (depending on the sample albedo), topography and imaging acquisitions can be performed simultaneously
Sample requirements	Samples should be extracted from the canister.
Effect on sample	None (generally, SNOM is a not invasive technique). However, during measurements the sample is in very close proximity to probe tip so risk of contact is high with the possibility of contamination and/or disruption of sample, especially in the case of friable materials. High sample stability requirement may entail working with fixed/secured samples that result in significant contamination and modification.
Links	Optical imaging, 3D imaging, electron microscopy
Data output	Multiple MB (topography and optical) images per sample
Service requirements	Lab electric supply
Examples	The Witec Alpha300S is a typical advanced SNOM microscope. Options include combining with Raman for high spatial resolution Raman.
Other info	SNOM technique can be coupled with spectroscopic techniques in order to obtain near-field spectroscopy. Spectrally resolved SNOM is possible also by considering a tunable light source

4.2 Methods for Characterisation of Physical Properties

HIGH PRECISION BALANCES	
Principle measurements	Measure mass of samples
Instrument specifications	Range of analytical balances required – Precision balances – for large samples – e.g. assess total mass returned or present in a large container. Capable of measuring several hundred grams with a precision of ≈1 mg. Semi-micro-balances – for individual specimens – up to 200 g, with 10 micro gram repeatability. Minimum sample size of the order of 10 mg. Work-horse balance, with high volume, high throughput of samples - so easy operation (touch screen) and fully integrated to central sample data archive to minimise work load Micro-balances = for small specimens where required/applicable. Mass range from 0.1 mg to gram, with repeatability at the microgram level. Fully automated with touch screen and automatic data archiving.
Sample requirements	Sample needs to be in container with accurately known weight
Effect on sample	None – balances need to work inside glove boxes or clean rooms. Verification required as to compatibility of balances with clean room and glove box operation. Automatic draft covers readily available – but for Precision and Semi-micro-balances these covers can be very bulky.
Links	See examples
Data output	Sample mass
Service requirements	Stable power supply, thermal environment and low vibration and air movement.
Examples	There are several manufacturers producing ranges of balances with similar capability. The following examples are from Sartorius. Precision balance – Sartorius Secura Semi-micro-balance – Sartorius Cubis Micro-balance – Sartorius Cubis MicroBalance
Other info	Technology development – would be good to assess what options available for robotic delivery of samples to balances exist, and their compatibility with the specific requirements of the clean rooms and glove boxes in the curation facility.

To determine the shape, internal structure, mineralogy and perhaps even mineral chemistry of samples without disruption of the sample.

X-RAY COMPUTED TOMOGRAPHY	
Principle measurements	Determining shape and internal structure of fragments up to cm or even several cm with a voxel resolution down to micron scale.
Instrument specifications	High energy micro-focus X ray source (e.g. >200 kV, ≈micron focal spot size at >3W) – permitting micron resolution of features in large (≈cm sized) samples. Capable of accommodating large samples (up to typical core diameter (<1 cm) and length (≈6 cm). Voxel size increases with increasing sample size generally – e.g. 8 cm samples with voxel size ≈50 micron. Sample stage travel to accommodate samples and stage rotation
Sample requirements	Samples should be analysable in sample container.
Effect on sample	None, although there is a need to secure the samples during measurements to prevent movement and therefore potential for contamination from contacting surfaces and mechanical damage exists.
Links	Optical microscopy, 3D imaging, X-ray diffractometer
Data output	Large data files – up to TB per sample
Service requirements	Un-interrupted single phase clean power supply
Examples	Zeiss Xradia 520 Versa is an example of the latest generation x-ray microscope. Capable of imaging large samples, and deep sub-micron spatial resolution..
Other info	This is a relatively immature technique when applied to the curation of extra-terrestrial or other contamination-sensitive geo-materials. A number of areas have been identified that required further development and/or characterisation. Secure sample positioning without contamination needs to be addressed. The samples are analysed inside the instrument, and therefore to minimise contamination the samples should also remain inside clean sample containers (ideally multi-walled) prepared within the controlled environment of the curation facility. The sample container should also have low atomic density, and distinct from samples. So while quartz may be considered the cleanest container material, there is limited contrast with typical rocky samples. Carbon-rich materials are generally preferred for many studies, but this is a potential source of contamination. Long duration measurements involving rotating the sample requires that the samples are well secured to minimise sample movement. This essentially demands that some material compatible with measurements is in direct contact with the sample. At the present time a suitable material for sample holding/containment has yet to be identified – that can protect the sample from contamination, hold the sample securely in the instrument during analysis and provide good transmission of x-rays. Some technology development is required in this area. Given the long duration of the measurements, one approach may be to mount multiple samples into a single large container. Automated analyses could perform low resolution scans of the

	container to identify where each sample was located and then perform high resolution images of only the space occupied by the sample. Variants of the next generation of instruments should be able to provide energy spectrum for each voxel, which in principle can provide chemical information, particularly useful in determining mineralogy. While on-going technology development is occurring verification of compatibility with complex sample holders will be required. The long duration and relatively high energy of the irradiation does offer some potential for modification of samples –e.g. effects on fission tracks. A dedicated study of the effects of such measurements on suitable analogues of each of the main sample types is required. A second CT-scanner may be required for the Receiving Facility - to investigate the sample container before opening. Depending on the size of the container, a larger CT-scanner may be required – e.g. the XT H 320 can accommodate samples up to 60 cm across with a mass of ≈60 kg.

MICRO X-RAY DIFFRACTOMETER	
Principle measurements	Produces a diffractogram from a sample irradiated with a x-ray beam, from which mineral identity and modes can be estimated (e.g. fayalite content of olivine can also be estimated from cell volume)
Instrument specifications	Position sensitive micro XRD Bright x-ray source Range of detector and sample stage modules – permitting range of analyses: Analyses of small powder samples (~mg) in sealed containers to preserve sample/maintain bio-seal(?) Micro-spot analyses – mapping and individual feature - ≤50 micron spot 3D computed tomography (CT) at low spatial resolution (50 micron) – but useful for occasional investigation – could be useful for preliminary investigation of sealed sample containers to assess state of sample prior to opening. Needs to be able to accommodate large samples – e.g. MSR tubes (1 cm diameter x 6 cm long)
Sample requirements	In situ, wide variety of sample format (for example, powder, thin section, epoxy mount, rock, sealed sample containers)
Effect on sample	Non-destructive, although there is a need to secure the samples during measurements and therefore potential for contamination from contacting surfaces and mechanical damage exists.
Links	Optical microscopy, spectroscopy, X-ray CT
Data output	Diffractogram (counts as a function of 2Θ)
Service requirements	Single phase laboratory mains electricity
Examples	PANalytical Empyrean
Other info	Systems with replaceable modules with high precision positioning allow for great flexibility with the same equipment. Particularly useful if instrument only used occasionally in certain modes. If instrument used routinely, e.g. for characterisation of powders, automated sample introduction or batch analyses may be appropriate, for which several solutions already can be provided by suppliers.

4.3 Spectroscopic Methods

To determine the mineralogy and nature of organic material in a sample a combination of FTIR, Raman spectroscopy and bi-directional visible and NIR spectroscopy (the latter to be used to characterize larger samples, i.e. > 20 mg)

FOURIER TRANSFORM INFRA-RED MICROSCOPE	
Principle measurements	Inteferometry compares returned/transmitted light energy of the IR sample spectrum to the source spectrum.
Instrument specifications	Possibility to cover wavelengths from 20 to 8300 cm^{-1} Spatial resolution from 3 μm of 25 μm Spectral resolution from 0.5 cm^{-1} to 16 cm^{-1} Wavenumber reproducibility of order of 10^{-3}-10^{-2} cm^{-1} S/N from 1000 to 8000 Possible detectors: DLaTGS, MCT, InSb, InGaAs, FR-DTGS, $LiTaO_3$) Desiccated and sealed optics Cassegrain objectives (magnification from 4x to 15x) Acquisition duration (for a mm-sized sample) from 20 seconds to 5 minutes
Sample requirements	No sample preparation necessary – although polished samples beneficial. Sample may be analysed in sample container or through (thin) containment wall. Suitable (low contamination) IR transparent material (tenths of mm), such as CVD diamond, is required for containers or windows into containers.
Effect on sample	None
Links	Bidirectional spectroscopy, Raman spectroscopy
Data output	Inteferograms or IR spectra
Service requirements	Computer interface and software control (RAM: 1 GB; HD: 5 GB) USB connector Power Supply PC to control vertical/horizontal adjustment and angle of rotation
Examples	Jasco (http://www.jascoinc.com/spectroscopy/ftir-microscopes) Thermoscientific (http://goo.gl/nyHrvD)
Other info	Possibility to couple with attenuated total reflectance capability.

LASER RAMAN MICROSCOPE	
Principle measurements	Measure the Raman spectra of individual components of samples without sample preparation
Instrument specifications	High spatial resolution – obtain spectra down to spot size ≈300 microns to ≈1 micron Laser spot size from 1 μm to 200 μm Spectral resolution from 0.4 to 1 cm^{-1} Multiple incident laser wavelengths (from UV to NIR) Working distance >15 mm CCD detector (multiple detectors required for optimised detection across spectral range of source lasers). Objective magnification from 5x to 100x
Sample requirements	Analyse inside sample container
Effect on sample	Risk of minimal damage in case of large power of incident light
Links	FTIR microscopy, bi-direction spectroscopy
Data output	Raman spectra and images
Service requirements	Supply Computer (4 GB RAM) and software to control instrument and capture images
Dimensions (w x h x d cm)	Approx. 45 x 65 x 35
Examples	Horiba XploRA ONE
Other info	Some Raman systems can accept a remote microscope head connected to the spectrometer via a fibre optic cable – allowing measurements to be made in restricted areas (e.g. glove boxes) with minimal requirements, access and contamination of environment/samples (but potentially at the expense of spatial resolution and/or sensitivity).

This measurement provides valuable ground truth for remote, in situ and ground based observations of planetary surfaces. If the returned sample is very small and much of the sample is required to perform a measurement then, as the technique can be entirely non-destructive, it may be possible to consider analysing the sample within the facility for security and sample preservation reasons.

BIDIRECTIONAL VIS-NIR SPECTROSCOPY	
Principle measurements	Bidirectional reflectance spectroscopy
Instrument specifications	Spectral range from 0.3 to 2.6 μm InSb detector Spectral resolution from 2 to 15 nm Possibility to change incidence (-10° to 70°), emission (-70° to 70°) and phase (10° to 140°) Acquisition duration: 2 hours
Sample requirements	Sample container proper of the instrument needed Minimum sample mass: 20 mg Sample area from 1.5 mm to 10 cm
Effect on sample	None
Links	Micro FTIR spectroscopy, Raman spectroscopy
Data output	KB ASCII spectra
Service requirements	Power Supply Computer and software
Examples	RELAB (http://www.planetary.brown.edu/relabdocs/RelabManual2006a.pdf)
Other info	

4.4 Electron Microscopy

To characterise samples at scales beyond optical resolution, and to provide chemical and mineralogical information electron microscopy should be used. This approach is particularly useful to characterise particulate matter – whether curated samples or contamination collected on witness plates, washes and filters used for monitoring clean room environments in the curation facility.

SCANNING ELECTRON MICROSCOPE	
Principle measurements	To perform high magnification imaging of sample surfaces, witness plates and other surfaces (x300 to at least x500000). Provide initial chemical information at micron scale – as quantitative spot analyses and semi-quantitative or qualitative chemical maps.
Instrument specifications	Field emission electron gun capable of delivering high resolution electron beam to sample (~1 nm). Accelerating voltage from 300V to 30 kV Low vacuum mode and Environmental SEM mode may be considered if requirement to study insulating samples without coating or volatile rich samples – although the contamination of the sample from the gas used may be significant. Secondary electron detector. Additional SED for environmental SEM mode if fitted. Lens mounted backscatter electron detector. Energy dispersive x-ray spectrometer – large area silicon drift detector. This could be normal type fitted at low angle to sample surface – alternatively a large area detector could be fitted immediately below the pole piece of the electron column for maximum signal count rates, optimising measurements for low kV – thereby minimising impact on sample Cryo Cleaner to maintain good vacuum in analysis chamber – in order to minimise sample contamination, particularly electron beam deposition of carbonaceous gases. Air lock for sample introduction – in order to preserve vacuum and therefore sample.
Sample requirements	A wide range of sample types and levels of sample preparation and processing can be analysed in the SEM: - No sample preparation or modification - Some samples can/will be as polished blocks or PTS - Some samples can/will be carbon or gold coated to aid charge dissipation Transfer system from clean room/glove box protects sample from contamination?
Effect on sample	Some electron beam damage to fragile materials on sample surface (outer few microns (primarily organic material, volatile-rich minerals). Some electron beam deposition of volatiles in SEM vacuum system – carbon-rich layers (nanometres) deposited on surface where electron beam rastered over surface. Localised (few micron) heating, devolatilisation where spot analyses performed. Coating on the sample (C or Au), that can mainly but not totally be removed by re-polishing samples if on polished sections.
Links	TEM, optical microscopy
Data output	High resolution images – up to several 10s Mbytes per image, many images generated per hour possible. Also small data files for quantitative elemental analyses. Proprietary software usually merges both data sets into integrated reports.

Service requirements	Un-interrupted single phase clean power supply Temperature stability – typically in small range (±1˚C) with slow temp change. Low vibration – both through floor and through air. Compressed air for vibration system and pneumatic valves. Dry nitrogen for venting. Vacuum pump exhaust.
Examples	A typical analytical SEM is the FEI Quanta although several other manufacturers offer similar products. A typical standard energy dispersive x-ray spectrometer could be an Oxford Instruments X-Max with large silicon drift detector, while the Bruker QUANTAX FlatQUAD offers exception sensitivity and rapid analyses, as well as the added benefit of high quality, low kV elemental mapping.
Other info	If SEM located outside clean rooms/glove box then sample transfer mechanisms can be readily developed that permit transfer of samples under controlled conditions (temperature, vacuum or inert atmosphere) – such as the Leica-Microsystems Vacuum Cryo Transfer System

Only required for specific sample types where the samples of interest are predominantly very small. Such sizes could be of the order of 10 μm, but may vary with sample type. The instrument is also required to prepare electron transparent (≤100 nm) for use in an TEM (see next page).

FOCUSSED ION BEAM SCANNING ELECTRON MICROSCOPE	
Principle measurements	Providing very high magnification images of the sub-surface regions of a sample by milling trenches. Preparing thin wafers of specific sample regions (generally < 20 x 10 microns) for other analytical techniques. Wafers can be electron transparent (<100 nm for rock) up to a few microns. The electron microscope aspects of the instrument are the same as for the standalone Scanning Electron Microscope described above. Unless extensive use is planned for FIB wafer preparation it is likely that a single instrument would suffice for most scenarios working with unrestricted category V samples.
Instrument specifications	Electron microscopy requirements as above. Focused ion beam, usually gallium – 300 V to 30 kV with current at sample adjustable from ~1 pA to >50 nA. Gas injection system (platinum organo-metallic complex) required for sample preparation and lift-out. Lift-out manipulator device – allows FIB wafers to be extracted from sample and mounted on suitable (usually TEM) mounts.
Sample requirements	For SEM measurements only – as above. For FIB work – insulating samples may require carbon or gold coating for charge dissipation. Can work with un-processed or polished samples.
Effect on sample	Carbon or gold coating required on insulating samples. Contamination, especially in the vicinity of the area studied, by platinum organo-metallic compound. Deposits of platinum-rich material around sample areas. Small holes (20 x 30 x 10 micron) where FIB sections prepared. Sputter debris around each sample area (includes platinum and organics from platinum deposit and gallium from the ion beam as well as re-distributed sample material).
Links	TEM
Data output	Same as for SEM
Service requirements	Same as for SEM
Examples	A typical analytical SEM is the FEI Scios Dual Beam Microscope, although several other manufacturers offer similar products.
Other info	Adding Scanning Transmission Electron Microscopy detector to the SEM is a small cost difference to the SEM, but would offer the possibility to verify quality of FIB wafers and micro-mill sections and provide relatively basic characterisation of samples at the sub-micron scale, potentially negating the need for a TEM.

Only required for specific sample types where sample of interest predominantly very small (typically ≤ 10 micron).

TRANSMISSION ELECTRON MICROSCOPE	
Principle measurements	Ultra-high resolution images of electron transparent sections and FIB-wafers. Provide mineralogical and chemical information ad <<micron scales.
Instrument specifications	200-300 kV electron column Magnification up to ~ x 500k Digital imaging Dark field and bright field detectors Energy dispersive x-ray spectrometer Electron energy loss spectrometer
Sample requirements	Samples must be electron transparent – typically <100 nm for silicates
Effect on sample	Extensive sputter damage to sample as a lot of material (relative to sample analysed) is removed to obtain electron transparent slices. However, the size of slices are generally very small – FIB slices generally <20 microns, ion milled sections could be over hundreds of microns. A contamination in Ga is observed on the main sample after the FIB slice has been extracted, likely related to the milling technique.
Links	SEM, FIB-SEM
Data output	Multiple high resolution images, spectra mads, data files. Could be 100s Mbs per analysis
Service requirements	Un-interrupted single phase clean power supply Low vibration environment Free from transient magnetic fields Excellent thermal stability (generally <±1 ˚C) Compressed air supply Vacuum pump exhaust.
Examples	The Hitachi H-9500 is an example of a standard TEM.
Other info	

For preparing sections a little larger (50 to 100 micron) than can be prepared by FIB

MICROTOMES	
Principle measurements	To provide electron transparent, or other very thin slices of small samples.
Instrument specifications	High level of automation to provide ease of use in glove box or clean environment. Accept diamond microtome blades required for fine grained rock samples.
Sample requirements	Samples must be very small (<100 micron typically), and mounted in resin blocks for sectioning (other mounting mediums possible (e.g. sulfur).
Effect on sample	Samples sliced to very thin sections Samples contaminated by mounting resin
Links	TEM
Data output	n/a
Service requirements	Clean stable single phase electrical supply.
Examples	The Leica Biosystems RM2255 Fully Automated Rotary Microtome provides the high levels of performance and repeatability required for precious and challenging samples.
Other info	

For preparing even larger areas of electron transparent samples, up to several hundred microns.

ION MILLS	
Principle measurements	To provide electron transparent, or other very thin slices of small samples.
Instrument specifications	High level of automation to provide ease of use in glove box or clean environment. Large area for milling Liquid nitrogen cooled stage to minimise sample artefacts during milling Variable argon ion acceleration from low (~0.1 kV) to high (~8 kV) to allow for range of materials to be processed.
Sample requirements	Samples prepared as thin wafers ready for reducing to ~100 nm thickness
Effect on sample	Samples sliced to very thin sections Samples usually contaminated by mounting resin
Links	TEM
Data output	n/a
Service requirements	Clean stable single-phase electrical supply. High purity argon gas supply Vacuum pump exhaust.
Examples	The Gatan PIPS II System provides the high levels of performance and repeatability required for precious and challenging samples.
Other info	

CARBON/GOLD COATERS	
Principle measurements	To provide conductive coat to samples for SEM and TEM measurements
Instrument specifications	Provide high purity, fine grained carbon or gold coat. Other coatings may be appropriate for certain samples. Accommodate samples up to 25 mm diameter. Expectation is that separate systems will be required for each coat type. Thickness monitor
Sample requirements	None thru to polished sections or electron transparent wafers/slices
Effect on sample	Sample contaminated by coating
Links	SEM, TEM, FIB-SEM
Data output	
Service requirements	Clean electrical single phase supply. High purity argon gas Vacuum pump exhaust.
Examples	The Quorum Technologies Q150T turbo-pumped Sputter/Carbon Coater is a good example of a easy to use coater for noble metals such as gold (Q150T-S) and as a carbon coater (Q150T-E). More advanced coaters, with ability to produce finest grain size coats, exist such as Leica EM ACE600, that has added functionality as already designed for accepting sample transfer systems.
Other info	

4.5 Chemical and Other Destructive Techniques

Primarily for monitoring and characterising contamination in Curation Facility.

TIME-OF-FLIGHT SIMS	
Principle measurements	Provide high sensitivity elemental analysis of surfaces. Primarily for monitoring contamination on witness plates and other surfaces in the SRCF.
Instrument specifications	High mass resolving power TOF MS (mrp ≥13000) Large mass range – up to ~200 Multiple primary ion guns – high spatial resolution Bi and metal cluster (gold) Accommodate typical witness plates (~ 200 mm?).
Sample requirements	No sample requirements – other than must be UHV compatible.
Effect on sample	Some sputter damage to surface – generally <1 micron over area analysed. Some sputter debris around area analysed
Links	SEM, TEM
Data output	Large image/mass spectra files – up to several hundred Mb per measurement
Service requirements	Un-interrupted single phase clean power supply High level of thermal control (±1 ˚C) Low vibration levels. Compressed gas Vacuum pump exhaust
Examples	An example of a high performance TOF-SIMS is the IonTOF TOF.SIMS 5
Other info	

A standard bench top GC-MS is sufficient for supporting clean room operations – characterising rinses from various cleaning procedures and swabs of surfaces. More advanced GC-MS such as GC-TOF-MS or GS-MS-MS may be required for Planetary Protection analyses.

GAS CHROMATOGRAPHY-MASS SPECTROMETRY	
Principle measurements	Characterising volatile and organic components in residues from rinses from various cleaning procedures, swabs of surfaces and solid and extracts of various analogue samples.
Instrument specifications	High temperature GC oven, with ability for multiple (2-3) sample introduction systems (e.g. split/split-less injection, variable temperature thermal desorption), liner, etc Quadrupole mass spec – electron impact ionisation; mass range up to ~1000 amu, ; full spectra to single ion monitoring. Robotic sample introduction system – capable of handling fluids, liners, automated extraction, etc.
Sample requirements	Sample should be reduced to concentrated solution prior to injection into GC. Principally for use supporting operations - small chips of analogue samples, witness plates, rinse solutions, etc. Potential use for samples – either solvent extracts or chips, powders.
Effect on sample	If returned samples analysed– either solvent extracted or heated to high temperature (up to >800˚C)
Links	LC-MS, ICP-MS
Data output	<few MB per analysis
Service requirements	Compressed gas lines for pure He (and possibly H_2 depending on final detector). Compressed air Vacuum pump exhaust. Un-interrupted single phase clean power supply
Examples	GC – Agilent 7890B MS – Agilent 5975C Robotic Sample preparation system – PAL RTC
Other info	Recommend additional screening system of GC with multiple simple detection systems (e.g. FID, TCID) with autosampler (e.g. Agilent 7693) for screening samples, detection of large/dirty samples etc.

LIQUID CHROMATOGRAPHY-MASS SPECTROMETRY	
Principle measurements	High sensitivity compound identification. Provides analysis of a complementary, and broader range of compounds than GC-MS.
Instrument specifications	Several variations of LC and MS can be combined to a LC-MS system. A robust system suitable for a wide range of complex samples (witness plates, rinse solutions, samples, sample extracts, etc). The most suitable combination for general use in the SRCF would be an LC system connected to a high mass resolving power Time-of-Flight mass spectrometer with electrospray ionisation. Detection sensitivity – femto gram Mass resolving power - >40000 Mass accuracy – ppm level
Sample requirements	Sample should be reduced to concentrated solution prior to injection into GC. Principally for use supporting operations - small chips of analogue samples, witness plates, rinse solutions, etc. Potential use for samples – either solvent extracts or chips, powders.
Effect on sample	Solvent extraction of samples
Links	GC-MS, ICP-MS
Data output	<few MB per analysis
Service requirements	Compressed air Vacuum pump exhaust. Un-interrupted single phase clean power supply
Examples	One example of a suitable LC/MS system comprises: 1290 Infinity II LC Systems Agilent 6550A iFunnel Q-TOF LC/MS
Other info	

INDUCTIVELY COUPLED PLASMA MASS SPECTROMETER	
Principle measurements	High sensitivity elemental analysis covering most of the periodic table. Primary use is for monitoring contamination on witness plates and rinse solutions.
Instrument specifications	Large mass range to cover periodic table – up to mass ~300 High sensitivity – ≤ ppb detection limits
Sample requirements	Sample for analysis must be a solution.
Effect on sample	Sample destroyed by dissolving.
Links	GC-MS, LC-MS
Data output	Small data files for each analysis – few hundred kbyte.
Service requirements	Un-interrupted single phase clean power supply Good thermal stability in the instrument and in the room Compressed air High purity argon gas supply Vacuum pump exhaust
Examples	An example of a standard high resolution ICPMS is the ThermoFisher Scientific Element XR ICP-MS
Other info	

X-RAY PHOTOELECTRON SPECTROMETER	
Principle measurements	X-ray photoelectron spectroscopy (XPS) surface analysis instruments provide elemental and chemical state information by measuring the binding energy of photoelectrons that have been excited with a mono-energetic x-ray beam. The principal application would be for the characterization of contamination deposited on witness plates exposed in the SRCF.
Instrument specifications	Perform XPS from large area down to ~10 microns Accommodate large witness plates (~200 mm diameter)
Sample requirements	None, although primarily for large, flat samples
Effect on sample	None
Links	TOF-SIMS, SEM, TEM
Data output	
Service requirements	Un-interrupted single phase clean power supply High level of thermal control (±1 ˚C) Low vibration levels. Compressed gas Vacuum pump exhaust.
Examples	PHI's X-tool is a good example of a simplified, user-friendly automated XPS system.
Other info	

ELEMENTAL ANALYSER MASS SPECTROMETER	
Principle measurements	Determine the elemental abundance of H, C, N on witness plates, and other materials
Instrument specifications	Sub ppm levels of sensitivity Expectation is that two instruments required – one set up for H and N, the other set up for C
Sample requirements	Witness plates and other samples required to fit inside small volume – suggest witness plates made of high puity aluminium foil.
Effect on sample	Sample combusted at high temperature (1000 – 1600 ˚C)
Links	GC-MS, LC-MS, ICP-MS
Data output	Small data files
Service requirements	Compressed air Compressed high purity gases Low vibration level Good thermal stability (< 1 ˚C) Vacuum pump exhaust.
Examples	Horiba EMIA-V2 series Carbon/Sulfur Combustion Analyzer for C measurement Horiba EMGA-920 H and N Analyzer
Other info	

4.6 General Laboratory Facilities

In order to support the analytical instrumentation some additional facilities are required on site.

Chemistry Laboratory Support:
In order to facilitate the maintenance of instrumentation a small chemistry lab is required for some aspects of cleaning and preparing parts of the system, particularly important for those instruments with vacuum systems, where high levels of cleanliness are required for all components inside the vacuum systems. If the instrumentation is located within a clean room environment then this chemistry laboratory should also be situated in a comparable environment. This facility should be equipped with various standard items of laboratory equipment including:

- a fume cupboard for handling organic solvents .
- a large ultra-sonication bath capable of holding items up to ~300 mm
- a drying oven, capable of reaching 200 ˚C and accommodating several items up to ~300 mm
- storage for glassware to be used for cleaning components and tools
- vented storage for a range of organic solvents (chlorinated and non-chlorinated)
- sink with hot/cold water for general cleaning.

For ICPMS a dedicated small chemistry laboratory is required for sample preparation. This should be located immediately adjacent to the ICPMS instrument lab. This chemistry lab must be a high level clean room (Class 100, with careful attention to materials – usually low VOC, metals) to minimise contamination of the samples (primarily witness plates). This lab requires:

- a large fume cupboard for sample acid digestion
- microwave acid digestion system
- a large ultra-sonication bath capable of holding items up to ~200 mm for cleaning tools/containers etc
- a drying oven, capable of reaching 200 ˚C and accommodating several items up to ~200 mm
- a hotplate resistant to corrosion
- storage for containers and tools
- Teflon labware
- vented storage for a range of acids
- ultrapure water supply (could use bottled water)

For the gas chromatography/liquid chromatography mass spectrometry laboratories, a dedicated sample preparation chemistry laboratory will be required. The prep lab will need to be of a high clean room level (Class 100) in order to minimise sample contamination. The lab is primarily required for solvent extraction of witness plates and concentration of rinses and extracts prior to analyses. The equipment and facilities required in this lab will include:

- a fume cupboard to handle organic solvents
- vented storage for a range of organic solvents
- an ultra-sonication bath capable of holding items up to ~200 mm for cleaning tools/containers etc
- a drying oven, capable of reaching 200 ˚C and accommodating several items up to ~200 mm
- a hotplate resistant to corrosion
- storage for containers and tools
- ultrapure water supply (could use bottled water)
- soxhlet extractor and super-critical fluid extraction
- ultra-centrafuge

SEM Sample Preparation
For large sample collections, preparation of polished thin sections and polished blocks a dedicated sample prep lab will be required. An extensive suite of tools and facilities are required. This is generally a process that creates considerable amounts of debris, and therefore careful consideration is

required as to the location of this facility relative to other areas of the SRCF as well as the layout and use of this facility to eliminate cross contamination of samples. In a facility with multiple collections, separate sample prep labs may be most appropriate. Equipment required will include:

- High precision diamond wheel saw (for larger samples band saws may be more appropriate) – e.g. Struers SECOTOM-15.
- Sample grinding and polishing systems, able to work with a sequence of abrasive grades – best if separate machines used for each grade (requires 4-5 machines?). Example of type of equipment is the Struers LABOSYSTEM.
- Optical microscope (petrographic with polarizer/analyser) to monitor sample preparation progress
- Sink with clean hot/cold water for cleaning equipment, tools and samples between stages.
- Ultra-sonication bath to clean tools, samples(?), containers, etc.
- Drying oven capable of reaching 200 ˚C
- Fume cupboard for handling organic resins and solvents
- Large area hot plates for sample curing
- Storage areas for materials, reagents, resins, tools, consumables, etc.

The location of this facility should be outside any clean room environment as the entire process can generate huge amounts of particles. This is particularly sensible when applied to large samples. However, when samples mass is very limited and/or particles are very small (e.g. Stardust, Hayabusa) then sample preparation in clean environments is more important, and because of the reduced sample mass being processed, more viable. In such a scenario specialist high-precision sample preparation equipment such as the Leica EM TXP Target Surfacing System for cutting, grinding and polishing under constant observation and high levels of automation could be installed in individual extracted clean glove boxes.

General laboratory equipment and facilities required to support instrumentation required – particularly for the destructive analyses. This includes chemistry laboratories with clean room/fume extraction, solvent extraction equipment, distillation set ups to produce ultra-pure reagents, polished mounts and thin sections, etc.

Location of Analytical Laboratories

While the priority and usage of each instrument may vary between the different sample types under consideration for this study some general rules can be considered.

Optical microscopes, balances and optical spectroscopy instruments (Raman, Vis-NIR spectrometer, FTIR) should be located within the cleanest regions where the samples are processed as the basic information collected, while important for curation yields little science return and therefore should be collected with minimal impact on the samples. Where it is deemed inappropriate or too challenging to accommodate the instrument within the clean area, the labs should be constructed to permit measurements to be performed through the walls of the clean room environment. This also applies to x-ray CT.

SEM, FIB-SEM, TEM and TOF-SIMS together with various coaters and sample thinning equipment should not be located within the clean room environment for several reasons. The samples will likely experience some modification during the measurement process, albeit this could be minimised to a very low level if the systems are well maintained and operated and therefore cannot be considered pristine after such analyses. The instruments themselves are rather incompatible with clean room environment, with many moving parts and made with materials often considered not ideal for clean rooms. These complex instruments require routine and frequent maintenance, usually requiring engineers from the manufacturer to attend who may not be familiar with the all protocols related to clean room work. However, sample transfer technology is mature and therefore, in the case of samples that do not present any bio-hazard, moving samples between clean environment and instrument can be readily performed without any additional compromise of the sample. Location of

all these instruments would be appropriate, as most of their service requirements are similar, and they can share the support equipment and sample preparation in most cases.

The elemental analyser, LC- and GC-mass spectrometers should also be located outside the clean environment, for many of the same reasons as for the electron and ion beam instruments, as well as the fact that these instruments will be primarily for clean room operation support rather than analysis of samples.

The floor area for each instrument has not been determined. However, in order to maintain clean environments, reduce noise, eliminate potential for mis-labelling of samples, and cross contamination each instrument should be housed in an individual laboratory or area. Within each lab, the total floor area required will be dominated by circulation space and operator work station, and the relatively modest foot print of the actual instrument (range by factor of approx 2) means that there will be little variation in the room size required. Most instruments should be comfortably accommodated in a 20 – 25 m^2 lab (TOF-SIMS and XPS may require slightly larger). Smaller equipment items such as balances and optical microscopes require only small areas of bench space (typically <0.5 m^2) and therefore can be readily accommodated more or less anywhere within the facility that they are required, including clean rooms, glove boxes, etc.

Staffing

The instruments described in this report all demand some level of maintenance from time to time, with the larger, more complex instruments requiring considerable levels of support just to remain switched on. The following defines the minimum staff effort required to maintain the suite of instruments in a state of readiness such that they can be used with little notice, for the operators to undertake some light, infrequent use of the instruments and to provide some training of other users.

For the optical microscopes and balances, low levels of maintenance are generally required, and therefore on the assumption that there are only modest numbers of each of them it would appear reasonable that staff involved in the general support of the clean rooms and/or samples could maintain these items alongside their main dutoes **(0 dedicated staff)**

The Raman, FTIR and vis-NIR spectrometer all share many similar characteristics, and requiring relatively limited maintenance one skilled operator could maintain this spectroscopy suite. **(1 staff)**

The SEM, FIB-SEM and TEM are all sufficiently similar instruments that it is reasonable to assume that a single operator could maintain these three instruments. The TOF-SIMS is quite different, and likely require a separate operator, although it would be reasonable to assume that this person could be trained to a sufficient level to assist the electron microscope operator. Both staff required maintaining this facility would need to be highly skilled. **(2 staff).**

The x-ray CT is very specialised compared to the other instruments, particularly demanding to support data processing and reasonable to assume would require a dedicated support person. The x-ray diffractometer, and XPS, while very different instruments, may have sufficient overlap to be maintained by one skilled operator. **(2 staff)**

The ICPMS is distinct from the other instruments supporting the facility, and likely requires specialist support. However, this is potentially not a full time position, and therefore the support and operation of the instrument could be readily undertaken by one skilled operator. **(1 staff)**

The LC- and GC-MS are quite similar in many respects, and therefore can be readily maintained by a single operator. The elemental analyser shares some common underlying technology/approach, and therefore it may be possible for a skilled LC-/GC-MS operator to maintain the elemental analysers as

well. **(1 staff)**

Some in house expertise will be required for the sample preparation for the SEM/FIB-SEM/TEM/ToF-SIMS and also for the LC-/GC-MS and elemental analyser instruments, and therefore a minimum of 2 further staff are required here. **(2 staff)**

In order to maintain this suite of 13 instruments, plus sample prep and associated microscopes and balances a minimum of 9 staff are required.

At times of heightened activity, such as might be considered in the build-up to the return of a sample and in the initial preliminary examination phase for 1-2 years post return additional staff would be required to prepare samples and operate the instruments. While the exact additional resource may depend on the nature and amount of sample returned, it would seem reasonable to assume that the minimum would include sufficient staff to operate all the instruments simultaneously with at least a doubling of sample prep staff. This could readily increase the staff working in this area to at least 15. In order to minimise the training period required for any additional staff, a model could be considered where suitably skilled scientists from the research community are seconded to the SRCF, either for short periods or for the duration of enhanced activity. NB, this does not consider the staff required to processing and performing cataloguing of samples in glove boxes or clean rooms.

While the staff involved with the instruments may be expected to spend considerable time in the laboratory, they would also require some time in a suitable office area to document and plan work, prepare reports, etc.