EURO-CARES

A PLAN FOR EUROPEAN CURATION OF RETURNED EXTRATERRESTRIAL MATERIALS

WORK PACKAGE 4

SUMMARY REPORT

(DELIVERABLE D4.5)

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1. Introduction

Any return of extraterrestrial samples to the Earth and their analysis by up-to-date laboratory methods can be divided in several individual steps or tasks summarized in Fig. 1. Identification, evaluation and articulation of these tasks is the goal of the EURO-CARES program. This will serve as a basis for recommendations for future sample return space missions. The following steps need to be undertaken: (1) Upon landing of the sample return capsule, it must be recovered and brought to a dedicated facility referred to as the Extra-terrestrial Sample Curation Facility (ESCF). (2) In this facility, the capsule must be opened, while respecting bio-hazard procedures in the case of restricted samples and minimizing sample damage and contamination at the same time. (3) Some extent of characterization is required for identification and cataloguing of the samples before (4) they can be allocated to the worldwide scientific community. Notably, sample characterization is of particular importance, and can be subdivided into the Sample Early Characterization (SEC) and the Preliminary Examination (PE). The second is at the corner stone between the SEC and protracted scientific investigations, including life detection that will be performed in parallel.

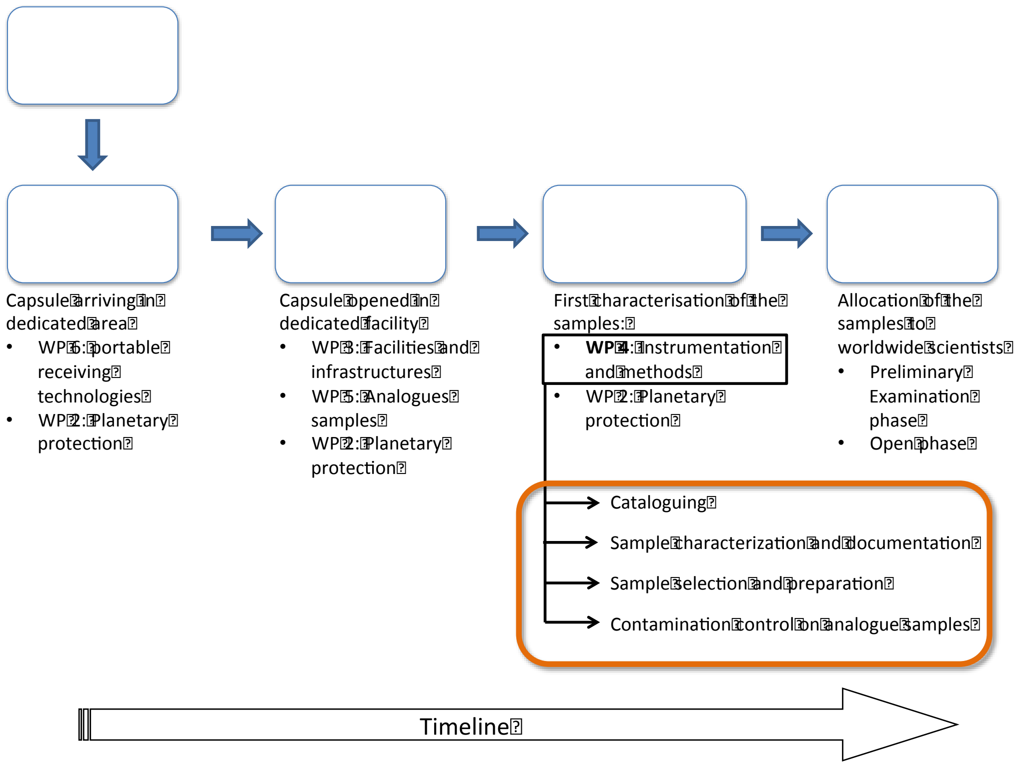

Figure 1: Summary of the different steps followed by the extra-terrestrial samples once arrived on Earth. The WP4, concerning the present final report, is highlighted and detailed.

The sample characterization task is the focus of Work Package 4 (WP4), Instrumentation and Methods. WP4 is more specifically in charge of defining which type of analyses are required and how they could be performed within the ESCF to optimize the sample characterization before scientific analysis.

The objectives of the WP4 were to:

(1) Build up on existing knowledge, including a review of literature regarding investigation and manipulation of extra-terrestrial samples, whether naturally arriving on Earth (meteorites, micrometeorites, interplanetary dust) or returned by space mission. This was the topic of D1.4. It also included the visit of existing curation facilities, as detailed in D4.1, and interactions with the scientific community. This was done in numerous occasions owing to discussions at international meetings and conferences, including the EURO-CARES meetings and more specifically the WP4 workshop held in Paris in 2016, as explained in D4.3.

Table 1. List of the WP4 deliverables

Deliverable	Title	Submission date
D1.4	Preliminary requirements	10/05/2016
D4.1	Space agency visits	21/11/2016
D4.2	Instrumentation	07/12/2016
D4.3	Workshop report	21/11/2016
D4.4	Industrial visits	18/05/2017

(2) Make an inventory of a number of possible techniques and methods, from very classical to cutting-edge technology, in order to determine which are priority, which are secondary, and to eliminate other (for example heavy, highly specific instrumentation such as SIMS). For that, several internal WP4 meetings have been held all along the duration of the program and the results were presented in D4.2.

(3) Once methods of choice are defined, evaluate corresponding instrumentation, conditions of operation and manufacturers as of 2017. This has been done based on our knowledge and personal professional experience, interactions with colleagues and contact with manufacturers, including more specifically those visited in the frame of the WP, and reported in D4.4.

(4) Raise questions for future developments by establishing a list of unanswered questions

The discussions and work have been led by WP4 keeping in mind several major questions regarding the aims and objectives of an ESCF. Those major questions are:

- - When does the sample early characterisation (SEC) stop? This is directly related to allocation and how the preliminary examination (PE) should work. As such, part of the SEC could be done within the facility, while other part could be externalized during the PE phase. This was the reason for distinguishing SEC and PE, even though there is a continuum between the two that will depend of external factors, i.e. the instruments available in the ESCF.

- - What are the boundaries between characterization of unrestricted samples vs biohazardous? This can be summarized as
  a) what are the instruments that are / are not required for both types of samples?
  b) is it possible to use the same instruments on both types of samples to minimize the costs and staff?
  c) is it possible to do both simultaneously?
  These particular issues required close interactions between WP2 and WP4 all along during the course of the EURO-CARES program.

- - How is instrumentation related to the infrastructure of the ESCF? In that respect a key question that has been discussed is how the requirements on instrumentation influence the number of facilities, i.e. if instrumentation requires to have all activities gathered in a same place or on the other hand if instrumentation favours separated places for the different activities (e.g. restricted vs unrestricted). This has been discussed in interaction with WP3.

These points are developed here with the recommendations of WP4 after the synthesis of the work done during WP4 activities.

2 Previous experiences

2.1. Workshop

The Work Package 4 (WP4) organized its workshop dedicated to the "Instrumentation and Method" in Paris on October 13th and 14th 2016 (Muséum National d'Histoire Naturelle MNHN). The main idea of this workshop was to summarize the previous experiments acquired by the different space agencies and/or laboratories involved in the curation and characterization of extraterrestrial samples. In addition, we also focused the workshop on the state-of-the-art techniques that could be important regarding the establishment of a European curation facility. The workshop was organized in two parts with the first one being restricted to the EURO-CARES members while the remainder of the meeting was open to the scientific community of planetary sciences in the broad sense. Fifteen EURO-CARES members were present for the restricted meeting and 38 persons attended to the open meeting (15 EURO-CARES members, 11 invited speakers and 12 other participants).

Specific attention has been paid to cover a large range of problems regarding the curation and characterization of extra-terrestrial samples. Hence, the WP4 workshop was divided into four main sessions:

1- A summary and an update of the WP4 activities since the Panel Review Meeting that took place in Paris in February 2016. Dedicated talks related to status of deliverables, the instrumentation list and the visits of the curation facilities (JAXA and NASA) were given by different members of the WP4 group. Other Work Packages also presented short updates of their activities with specific emphasis on those related to WP4.
2- Four invited speakers presented the actual situation of different curation facilities existing in the world today. These talks also addressed the procedures for storing and handling extra-terrestrial objects of different natures. Invaluable information was presented regarding (i) the organization and preliminary examination period for different NASA sample return missions (Mike Zolensky, NASA, Houston, USA), (ii) the benefits (and difficulties) of a cold-curation under controlled-atmosphere (Christopher Herd, University of Alberta, Canada), (iii) the conditions and the proposed protocol for future samples that will bring back to Earth by the OSIRIS-REX space mission in 2023 (Kevin Righter, NASA, Houston, USA) and (iv) the new curation facility recently opened for the storage and manipulation of micrometeorite from the CONCORDIA base in central Antarctica (Jean Duprat, CNRS, Orsay, France).
3- Four invited speakers presented analytical non-destructive techniques that are considered as key techniques by the WP4 group for future curation facilities. These characterization techniques of extra-terrestrial samples are related to: (i) the magnetic characterization (Jérome Gattacceca (CNRS, Aix-Marseille, France), (ii) the Fourier Transform Infra-Red characterization (FTIR, Rosario Brunetto, CNRS, Orsay, France), (iii) the X-ray Computed Tomography of samples (Natasha Almeida, NHM, London, UK) and (iv) the Time-Of-Flight Secondary Mass Spectrometry technique to monitor the surface contamination of samples (TOF-SIMS; Laurent Thirckell, CNRS, Orléans, France).
4- The last session was dedicated to presentations by invited manufacturers. Recently developed techniques were presented that could be important for future facilities: (i) Focal Plane Array detectors for FTIR (Agilent), (ii) 3D microscopes (LEICA) and (iii) X-ray microscope and correlated microscopy tools (ZEISS).

A general round table followed these four sessions. The main topic was related to the extent of preliminary characterization to be performed and what is the limit between initial and detailed characterization. Other specific points were also discussed such as the need for automation, the presence of a synchrotron light source close to the facility and the question of the definition of a "non-destructive technique".

The discussions during the WP4 workshop were really helpful and specific important points have emerged. Apart from the Apollo and Hayabusa sample facilites, all the curation facilities that have been built (or will be built in the near future - Hayabusa 2 and OSIRIS-REX) were retrofitted from spaces initially dedicated to other uses (e.g., office spaces). Such a strategy significantly reduced the cost for the establishment of the facility from $10,000,000 to $100,000. This puts into perspective the budget requirements for the development of a new European curation facility. The question of the perimeter and the composition of the Preliminary Examination team animated the debate as it varies from one mission to another. Some space missions have adopted an operation mode open to volunteers (Stardust) while others will operate with a PE team consisting of a limited number of people. It should be noted that the Hayabusa space missions worked with two PE teams. In addition, the crash of the GENESIS space mission implied a specific cleaning procedure that increased the duration of the PE phase and the prerogatives of the PE team. Another important aspect is related to the monitoring and control of the contamination. The OSIRIS-REX mission made pre-flight experiments to ensure the minimum contamination level during the operation. The peculiar nature of carbon-rich (micro)-meteorites requires the design of specific facilities for preventing organic contamination (CNRS Orsay and University of Alberta) with recommending temperature operation at -15°C and temperature storage at -80°C. This requires the use of materials that meet the low temperature requirement. We concluded that TOF-SIMS represents the ideal machine for tracking potential surface contamination generated during storage. Considering all the discussion during the workshop, it appears that the PE of the Stardust mission can be considered as a reference with very low cost duration, efficient distribution and high scientific output. However, this protocol cannot be followed for biohazardous samples and the extent of work done inside the facility depends on the nature of the samples.

To summarize, the WP4 workshop was successful and generated stimulating discussions, which induce a lot of progress in a very limited amount of time. This allowed a first set of general recommendations to be drawn:

(1) Monitor contamination by keeping a record of materials and conditions during pre-flight spacecraft assembly and in curation facility.
(2) Develop a quick and efficient preliminary examination involving many groups outside the facility in order to be cost-efficient.
(3) A portable clean room installed at the landing site of the sample capsule would allow the management of the unexpected to be performed in good conditions.
(4) Instrumentation and methods must be adapted to individual missions depending on the science goals and sample type (including size)

(5) The extent of sample damage induced by different characterization methods including those usually considered to be "non-destructive" requires detailed cross-studies.

2.2. Space Agency visits

Several visits of space agencies with functional curation facilities have been undertaken the last two years. We focus on the two facilities that currently curate returned sample materials: the National Aeronautics and Space Admiration (NASA) and the Japan Aerospace Exploration Agency (JAXA).

Hayabusa-returned sample curation facility (Kanagawa, Japan)

The Planetary Material Sample Curation Facility (PMSCF) was completed in March 2008 for the purpose of the Hayabusa 1 space mission that collected grains at the surface of the asteroid Itokawa. The PMSCF was built from scratch and integrates a comprehensive plan for rooms of the curation and a public part for outreach activities. The curation facility is located in the JAXA building complex, close to the control room of the spacecraft Hayabusa 2, thus allowing direct interventions *24 hours a day*, seven days a week, 365 days a year. Operating the clean chambers requires a lot of practice. The curation facility consists of a garment room, leading to four main clean rooms with different levels of cleanliness for a total surface of 400 m^2. Two clean chambers mainly made of stainless steel 304 are used to store and handle the samples. They are equipped with state-of-the-art vacuum system, optical microscopes, cleaning tools and micromanipulation systems. These chambers are kept under positive nitrogen pressure relative the atmosphere with chamber #1 being equipped with a system providing purified evaporated liquid nitrogen. This system allows the contamination to be minimized. Specific people from JAXA were selected and trained for this difficult job. The sample transfer from the clean chamber to the sample holder is performed by two people according to a very detailed protocol. The samples located on quartz glass disk are first observed and photographed by two optical microscopes set in the system. Then, the samples are picked up using a micromanipulator system that is composed of a needle on which a voltage is applied. This allows samples to be grabbed using static electricity and transferred to the sample holder for characterization. This part of the process is extremely difficult and requires perfect training.

It should be noted that some of the non-cleanrooms were designed to be easily retrofitted for the Hayabusa 2 space mission (currently in progress). Although Hayabusa 2 will be focused on volatile, organic matter and noble gas investigations, JAXA considered that the present curation facility has sufficient performance for curation of these precious extra-terrestrial samples. Except for some minor modifications, the JAXA will design a similar curation facility for Hayabusa 2 samples.

Lunar Sample Laboratory Facility (Houston, USA)

This facility was built in 1979 to host 382 kg of lunar rocks, cores and soils brought back by the Apollo missions. The complex is composed of a large curation room, a large vault, a laboratory for inviting researchers working on disseminated samples, and a smaller vault for returned samples. The storage and curation are performed in stainless steel glove boxes under a dry nitrogen atmosphere flow. Inside the glove boxes, specific instruments and tools are available allowing manipulation and splitting of the samples. Samples are handled to prepare sub-samples for scientific purpose dissemination, which are weighted and stored on aluminium tag. The access to the sample curation facility is restricted to a limited number of people.

Genesis Processing and Sample Storage Facility (Houston, USA)

This curation is a retrofitting of a part of the Lunar building (former visitor part) and is composed of successive rooms of increasing cleanliness. The main operation is related to the characterization and catalogue the Genesis samples and to disseminate the sample to research institutions. Few instruments are used inside the facility (mainly FTIR microscope).

Stardust Laboratory (Houston, USA)

This laboratory is a retrofitted room of the Lunar Laboratory composed of a curation and a storage room. The samples and the aerogel sample dust collector tray are stored inside a stainless steel cabinet flooded with pure N_2. Specific attention is paid to the humidity levels as the aerogel is really sensitive to water alteration. Instrumentation is very limited with only microscopes, video cameras and micromanipulators. The curation room is equipped with anti-static floor and the sample extractions are performed on vibration isolation tables.

Cosmic Dust Laboratory (Houston, USA)

This facility hosts a collection of cosmic dust from 1981 to the present day. The laboratory is composed of a room for instrument storage and another for the curation and preliminary examination (ISO 4). As no detailed characterization is performed in the laboratory, the number of instrument is thus limited with binocular microscopes and micromanipulators.

3. Methods

The primary function of the sample curation facility is to preserve returned samples in a pristine condition, and a second high-priority function is to provide samples to the scientific community. Multiple, potentially conflicting requirements exist in relation to this aspect of the work that include:

1) providing a detailed catalogue of the samples
2) providing sufficient characterisation of the samples to allow identification of the most appropriate ones for scientific requirements.
3) providing sub-samples of specific samples with minimum loss and modification
4) providing a record of key contamination markers that the samples are exposed to during processing and storage

The simple storage of the samples in the curation facility can result in some level of modification and/or contamination, as the storage environment almost always differs from that where the samples are collected, and the samples will undoubtedly be in physical contact with some form of storage container. However, it is likely that manipulation and processing of the samples to provide the information above in 1) to 3) has the potential to lead to more significant modification and contamination and therefore a suitable compromise is required to provide the optimum balance of level of information against the preservation of sample material.

Figure 2 (a) Polished thin section of a meteorite. Polished wafers of samples (typically 30 μm thick) are glued to glass disks.(b) Polished block of meteorite. A sample of material is mounted in a resin block, which is then ground and polished on one surface. The prepared surfaces have a high quality finish that is necessary for a wide range of microscopy and analytical techniques. Images courtesy of Wooddell.

The main aspects of each of these activities can be summarised as:

1) *Cataloguing*. This involves building a database that allows for the identification and record of each sub-sample that includes basic information such as written description, photo-documentation, potentially at multiple scales and in 3D, mass.
2) *Characterisation of the samples*. This covers a wide range of measurements; covering multiple aspects of the preliminary determination of the structure, mineralogy and organic inventory of the samples. The level of detail acquired is relatively limited as more detailed measurements of this type would be very time-consuming, require

extensive interpretation and extensive expertise and therefore the expectation is that such activity would be undertaken by the scientific community on allocated samples. Characterisation activities in the curation facility should be conducted with little, or no, impact on the physical and chemical nature of the sample.

3) *Sample selection and 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 (Figure 2), microtome section (Figure 3)) that the samples have been prepared properly.
4) *Contamination control and contamination knowledge;* This is a critical aspect of the sample curation process and includes monitoring of the clean room environment, and all cleaning and handling protocols that may impact the samples. Measurements may include direct analysis of gases or reagents used in the curation facility; the surfaces, or extracts of surfaces, of sample handling or storage devices and witness plates and test samples. Frequent measurements are required in order to verify that samples are not exposed to unacceptable levels of contamination and that cleaning and handling procedures are meeting specification. As contamination cannot be guaranteed to be zero, such measurements and witness plates will also provide knowledge about what contamination the samples are exposed to during their residence and processing in the facility, that will provide invaluable help in the interpretation of contamination sensitive measurements performed on allocated samples.

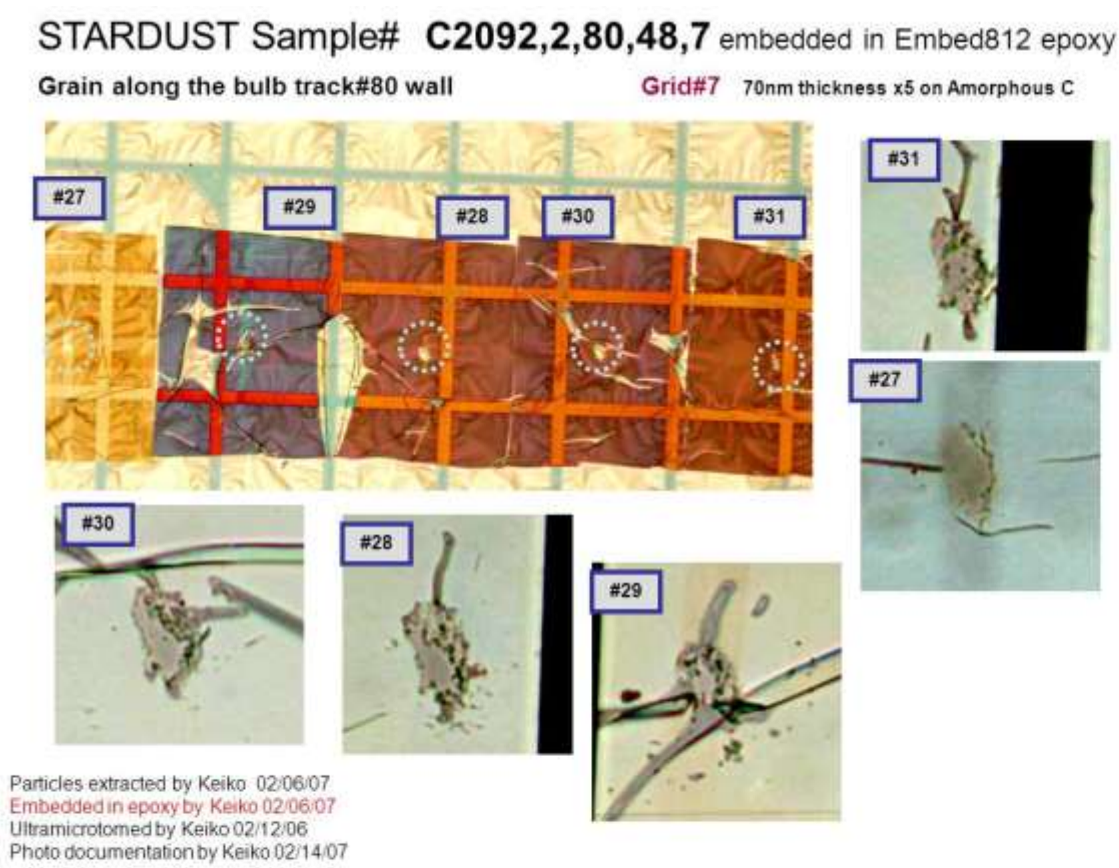

Figure 3 Microtome sections of Stardust mission cometary dust grains. The samples are mounted in resin and sliced with a microtome to produce wafers approximately 70 nm thick. Five sections shown, the rectangular slices of ca. 400 micron square contain the small cometary samples (circled and shown as separate images). Courtesy of NASA JSC Curation.

Table 2 shows the instrument priorities as identified in WP2 and WP4.

Instrument priority	**WP2**	**WP4**
Cataloguing		✓
Detailed microscopy and X-ray characterization		✓
Sample selection and preparation	✓	✓
Contamination control	✓	✓
Life detection and biohazard assessment	✓	

Table 2. Instrument priorities as identified in WP2 and WP4. It is anticipated that while there are differences in the priorities for the two activities, they would in fact be integrated for a restricted sample.

4. Instrumentation

The details of the cataloguing, characterisation, sample preparation and the most critical specific contamination types and levels are all mission-defined and unique to each mission or sample source, although for most large, rocky types of samples there is likely high levels of commonality. As such there is a broad suite of key instruments that can deliver all of the key documents, information and measurements. These instruments were identified and described in Deliverable 4.2: Instrumentation. Incorporating on-going visits to instrument manufacturers (see above), an updated version of the instrument requirements for an unrestricted sample curation facility is summarised here.

Instruments for Cataloguing/Documentation

Instrument	**Comments**
Low magnification microscopes	Multiple microscopes required
High magnification microscopes	Petrographic and materials
3D imaging/shape profiler	Technology development
High precision balances	Multiple required, large mass range
Scanning near field optical microscope (SNOM)	*Only for small samples (≤few micron particle size)*

All the main instruments here are all compatible with operation inside clean rooms or even inert gas clean glove boxes with either remote automated operation or through-wall operation.

Instruments for Sample Characterisation

Instrument	**Comments**
FTIR microscope	Spectral imaging detectors for rapid, high resolution
Laser Raman microscope	UV resonance Raman useful for organic analyses
X-ray CT (sample)	Separate X-ray CT for sample container
Micro X-ray diffraction	May be superseded by spectral X-ray CT advances
Analytical SEM	Multi-detector environmental SEM for insulators
Focused ion beam SEM	*Only for small samples?*
Analytical TEM	*Only for small samples?*

The FTIR and Raman microscopes can operate through clean environment walls, or possibly within clean environments without significantly compromising sample purity. X-ray

Figure 4 Sample transfer system produced by microscope manufacturer Leica (left) The samples can be loaded into transfer system under clean, inert atmosphere or vacuum conditions. The sample transfer device can then be attached to a compatible docking chamber attached to various compatible instruments and the sample transferred without exposure to unwanted contamination.

instruments and analytical SEM are not compatible with highest specification clean room environments, although sample chambers could be interfaced to clean environments directly with instrument primarily outside cleanest area, or samples could be transferred under sealed controlled inert gas or vacuum conditions from clean environments to sample chambers.

The FIB and TEM systems are only really required for samples with the smallest particle sizes. The nature of the analyses and the effects on samples negate any requirement for such samples to be operated in clean environments, although integrated sample transfer systems compatible with the analytical SEM would help minimise sample contamination and modification.

Instruments/Equipment for Sample Preparation

Instrument	**Comments**
Sputter coaters	C & noble metal (Au?) for SEM
Microtome	Primarily for small sample sizes
Ion Micromills	Primarily for small sample sizes
Micromanipulators	Electro-static tips, micro-tweezers for diff sizes
Integrated prep systems	High precision cut, grind and polish system
High precision saws	Diamond wheel (band saw for large samples)?
Grind and polish systems	Automated high precision systems for PTS

By their very nature, each of these instruments/tools creates debris that are to varying degrees are incompatible with operation in the cleanest environments that the samples are exposed to. Therefore, dedicated clean areas optimised for these systems are required to minimise avoidable contamination (some contamination and/or modification is unavoidable). Fully integrated systems exist which provide end to end advanced sample preparation (e.g. Leica EM TXP; Figure 4, 5) that are ideally suited to preparation of small, precious samples with remote/semi-autonomous operation and therefore compatible with clean bench operation. Further development to integrate with existing instrument transfer

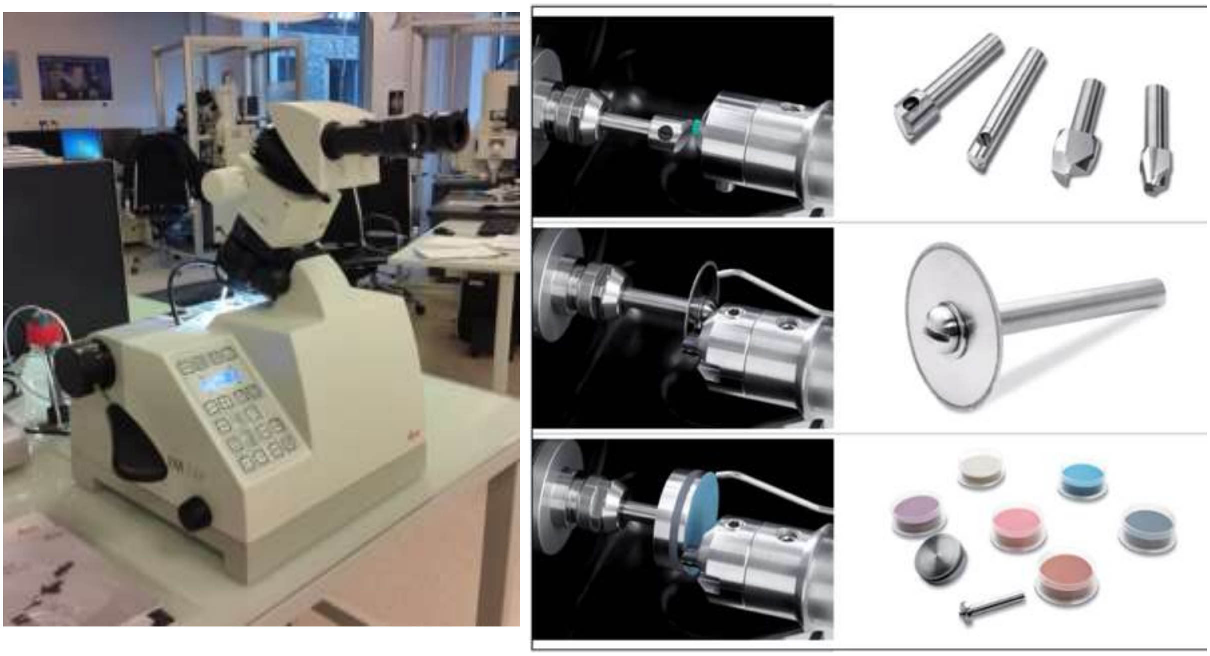

Figure 5 Leica EM TXP sample preparation system for polished mounts (left). On the right are some of the tools used for milling, cutting and polishing.

systems could ensure that a comprehensive integrated sample transfer system could provide protected preparation, transfer and analysis of all samples.

Instruments required for contamination control and contamination knowledge are shown below. The particle counters are required to monitor particle abundances in real time within the clean environments and residual gas analysers to monitor the abundance of trace gases and volatile organics in the clean sample handling and storage areas. These instruments are required with high frequency and will be an integral part of curation facility operations.
The other instruments are all required to assess the level and nature of the contamination at regular intervals (using witness plates to record contamination around key functions/samples, Figure 6), monitor the efficiency of cleaning protocols and handling procedures. The frequency and complexity of these measurements will be sample/mission specific. Samples/missions with high cleanliness demands may require frequent access with rapid turn-around time for results and it is therefore important for these instruments to be located on site within the curation facility.

Instruments for Contamination Control and Contamination Knowledge

Instrument	Comments
Time Of Flight-SIMS	Surface contamination in situ
Gas Chromatography-MS	Chemical characterization
Liquid Chromatography-MS	Chemical characterization
ICP-MS	Elemental abundances
X-ray Photoelectron Spectroscopy	Surface contamination in situ
Elemental Analysis-MS	C, N abundance
Residual Gas Analysis-MS	Continuous environment monitoring
Particle Counters	Continuous environment monitoring

As these instruments are not involved in the analysis of returned samples there is no need for such instruments to be in the main clean environment areas. However, to ensure that detection limits are compatible with the contamination levels expected in the cleanest areas dedicated clean rooms with protected sample transfer mechanisms are required. For less demanding sample cleanliness requirements the frequency of use of these may be relatively limited and therefore these analyses could be performed off-site under contract. As the planned sample curation facility is expected to host multiple mission with a range of requirements it is assumed that all these instruments would be within the curation facility.

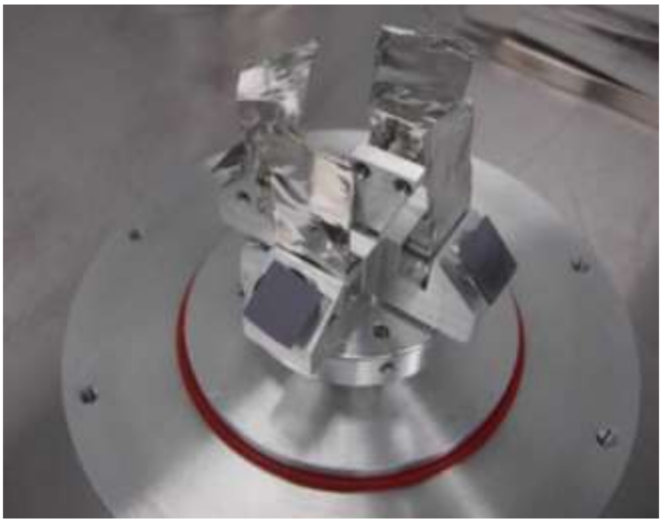

Figure 6 Witness plate assembly used to monitor the environment around the assembly of the OSIRIS-REx spacecraft. Aluminium foils were used to determine bulk organic content while the silicon wafers pre-mounted on SEM stubs were used to characterise particulate matter (Dworkin et al, submitted to Space Science Review (2017), http://arxiv.org/abs/1704.02517

5. Sample Preparation Facilities

Dedicated laboratories are required to support the operation of the instrumentation involved in sample processing, characterisation and contamination control.

Polished Sample Preparation
In case of collections of large rock samples, a dedicated sample prep lab will be required for preparation of polished thin sections and polished blocks. 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 ESCF 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.
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.

Chemistry Laboratory Support
Instrument Maintenance: 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.
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).
Gas chromatography/liquid chromatography mass spectrometry: 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 flow chart summarizing the four steps of sample processing and corresponding instrumentation for each step is shown in Figure 7.

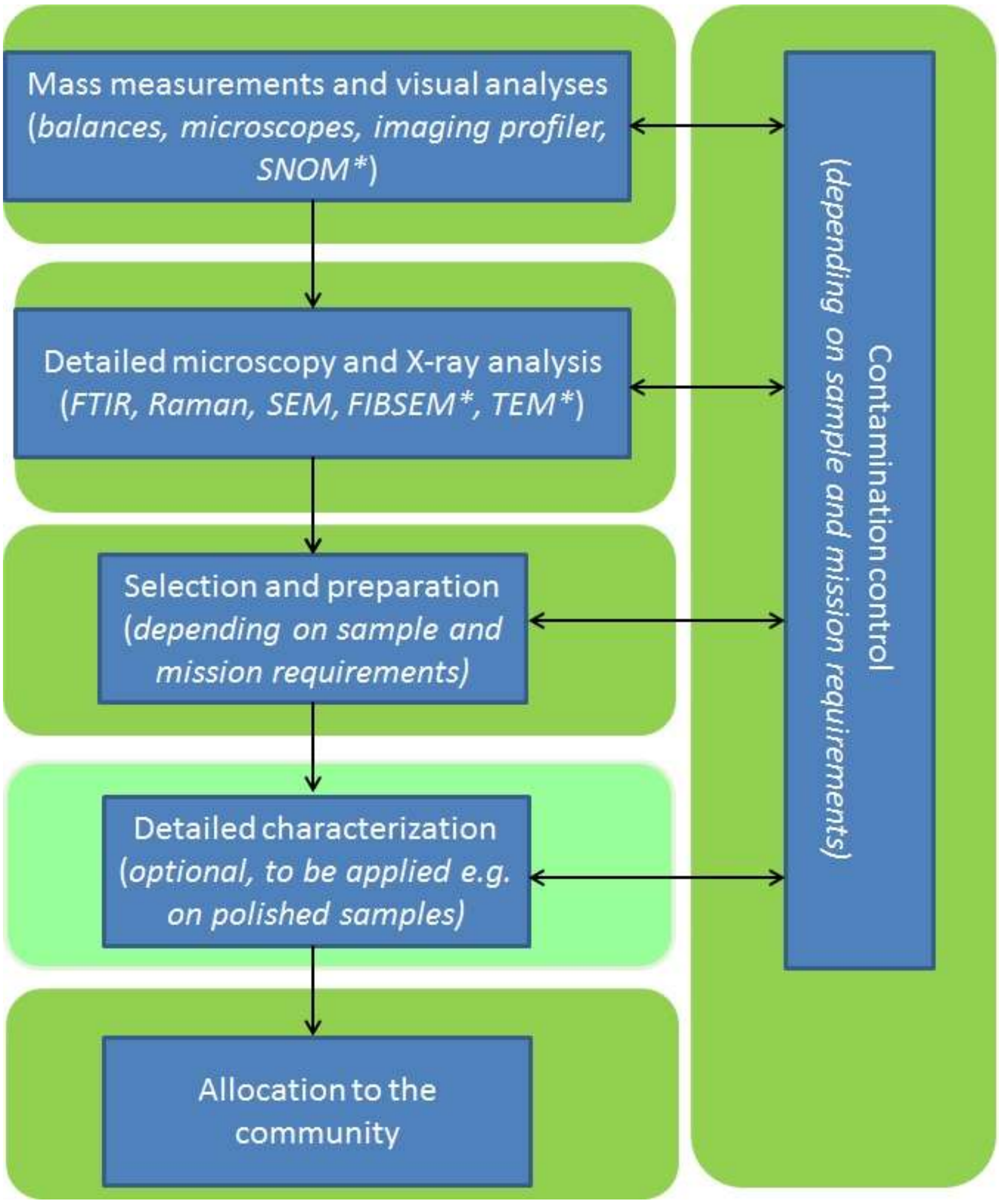

Figure 7 Summary of samples processing and identification of instrumentation for each step. Contamination knowledge is generally not associated with direct measurement of samples, but runs parallel to all aspects of sample processing.

6. Staffing

All the instruments required in the curation facility demand some level of maintenance, with the larger, more complex instruments requiring considerable levels of support just to remain switched on. An assessment of 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 was determined in Deliverable 4.2 and summarised here:

a) Optical microscopes and balances: low levels of maintenance are generally required, supported by staff involved in the general support of the clean rooms and/or samples **(0 dedicated staff)**
b) *Raman and FTIR microscopes:* share similar characteristics **(1 staff)**
c) *SEM, FIB-SEM, TEM:* sufficiently similar instruments so could be maintained by a single operator in principle. However this could be very demanding, and therefore a second operator required **(1 staff).**
d) *TOF-SIMS*: specialist support, but with some overlapping experience with SEM/TEM and therefore could also provide support to electron microscopy **(1 staff).**
e) *X-ray CT, X-ray diffractometer, XPS*: While three very different instruments, reasonable to expect sufficient skills and capacity from 2 skilled staff to support all three instruments. **(2 staff)**
f) *ICP-MS*: dedicated skilled operator required. **(1 staff)**
g) LC- and GC-MS: are quite similar in many respects, and share overlap with elemental analyser and therefore can be readily maintained by a single operator. **(1 staff)**
h) 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 12 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 post return the number of staff supporting and running instruments would increase. It was estimated in Deliverable 4.2 that at least 15 staff would be required, with the additional staff seconded from the scientific community. It should be noted that this does not consider the staff required for processing and performing cataloguing of samples in glove boxes or clean rooms.

7. Instrument visits

A deliverable of the WP4 was the visit of companies that propose generic products relevant to the storage and curation of extra-terrestrial samples of different natures. This includes sample handling tools, microscopes and microtomography. In order to optimize the different visits, a list of questions to be addressed was first established. These questions rely on the function of the instrument, its flexibility and how it can be operated in the specific conditions required by some curation facilities. In addition, we enquired about the cost and maintenance requirements. We detailed below the results of our visit to three main companies.

Agilent

The main activity of this company is to design and sell conventional gas/liquid chromatography, mass spectrometry products and Infrared (IR) spectroscopy. The latter (Topscan 4300) allows rapid characterization of large samples without introducing contamination. A more conventional IR microscope is also available but with new highly efficient Focal Plane Array (FPA) detector. This detector allows the simultaneous analyses of a sample surface by direct imaging using a defocused laser beam. This is interesting as it offers the possibility to acquire simultaneously a large number of spectra for a total acquisition time that is significantly reduced. The FPA detector has been developed by the US Army and is commercialized by only two companies including Agilent for a price of around $200,000. Agilent has experience of dealing with cleanrooms and has engineers with cleanroom habilitation. It should be noted that a new FTIR will be released in 2017 but it is still under commercial embargo.

LEICA

Leica is a German company with an optical microscopy branch. They propose different types of microscopes that can be easily customized for specific uses. In addition, optical microscope can be implemented with various types of spectroscopies (Raman, UV, IR). Leica also proposes software development that can be useful for sample handling, observation and image analysis. Interestingly, Leica is interested in analytical developments for specific scientific purposes. Leica also developed microscopes for very large samples and 3D microscopes that can be useful for curation facilities.

Leica commercializes a sample cutting and trimming system under stereomicroscope (EM TXP) that allows precision cutting and milling under vacuum to be performed. This system offers the possibility to prepare samples for observation with transmission electron microscopes or electron backscattered diffraction. This system has several advantages regarding the curation of extra-terrestrial samples: (i) precise cutting, (ii) handling small samples and (iii) limited dispersion of dust in the sample handling room. Leica also proposes a sample transfer system allowing sample transfer under vacuum at cryogenic temperature from an instrument to another. This tool would be highly advantageous in a curation facility, as it would reduce significantly the sources of contamination.

FEI

This company sells xenon ion plasma focused ion beam instrument (Xe-FIB). This emerging tool allows larger volumes to be excavated from samples than a conventional Gallium FIB. In addition, the use of a Xe-FIB reduces the contamination and the amount of irradiation damage to the sample from the beam. Considering the curation facility, such a technique would be helpful to prepare tiny dust grains with precise cutting in preparation for distribution to external researchers.

8. Summary of recommendations and conclusions

The work of WP4 produced a list of methods to be employed for the sample early characterization in the ESCF and of the required priority instruments to do so, of their conditions of operations, of the staffing required and of possible companies to contact to acquire, install and eventually customize these instruments. This was done based on instrumentation known as of 2017, but potentials for future developments were also taken into account, notably during interactions with manufacturers.

In addition to these lists, the discussions and interactions we had within WP4, within EURO-CARES but between different WP (mostly WP2) and with the scientific community, led to several recommendations pertaining to (1) the limit of the sample characterization and (2) the distinction between restricted and unrestricted samples in terms of instrumentation and analysis. These recommendations are summarized in Figure 8.

First, as the bioburden constraints are very different between restricted and unrestricted samples, the instrumentation required for both types of samples was evaluated separately by WP4 (focused on unrestricted samples) and WP2 (focused on restricted samples). We reached the conclusion that it is best to keep these analysis separated in the ESCF, in order (1) to avoid bio-hazardous cross-contamination issues, (2) to ease as much as possible the maintenance of instruments for the unrestricted samples. Keeping the two separated, eventually with duplication of similar instruments, allows working on unrestricted samples even if quarantine is required for different restricted samples. In the framework of the infrastructures evaluated by WP3, this corresponds to separated facilities, even though they can be located at the same place to optimize other issues such as outreach etc. Separated facilities in the same location also enable transportation efficiencies if early sample analyses indicate that samples must be transferred from one facility to the other.

Second, experience gained from previous missions and notably the Stardust mission, shows that the scientific output is maximal if the analyses in the ESCF are minimal and as rapid as possible. This is why we have split the initial analysis phase (of unrestricted samples) into two different steps: the first one referred to as Sample Early Characterization (SEC) corresponds to the minimal characterization as listed above and performed within the ESCF upon opening of the sample return capsule, by the facility staff. The second one, which is referred to here as the Preliminary Examination (PE) corresponds to the first scientific investigations aiming at reaching the missions' scientific goals. This can be done by science teams selected prior to the sample arrival on Earth or, as in Stardust, by including scientists applying on the basis of analytical or scientific

experience during the PE phase. After these two phases it can be anticipated that the samples will be available for the whole scientific community upon calls for proposals to answer different or unanticipated questions or to perform newly developed specific analyses.

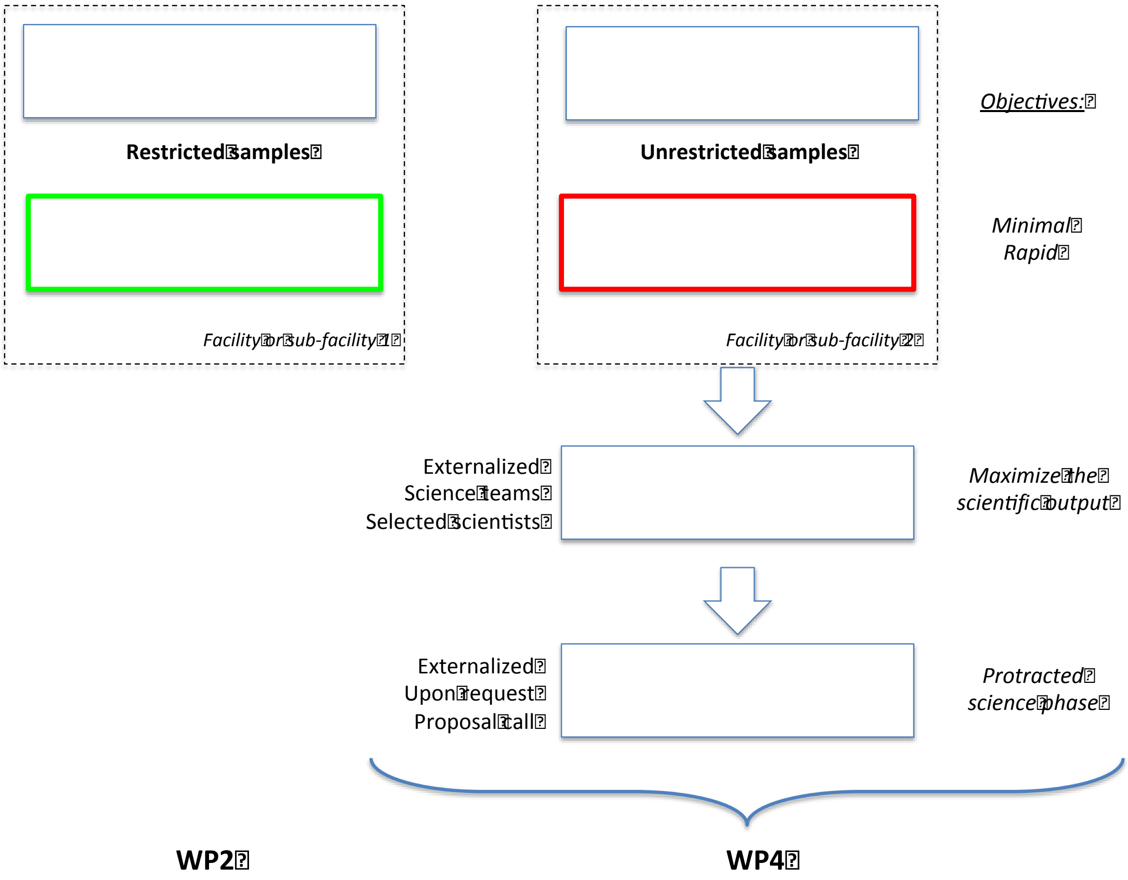

Figure 8. Summary of WP4 recommendations

Appendix

List of abbreviations

CNRS	Centre National de la Recherche Scientifique
CT	Computerized Tomography
Dx.x	Deliverable x.x
ESCF	Extra-terrestrial Samples Curation Facility
EURO-CARES	European Curation of Astromaterials Returned from Exploration of Space
FIB	Focussed Ion Beam
FPA	Focal Plane Array
FTIR	Fourier Transform Infrared Spectroscopy
GC-MS	Gas Chromatography–Mass Spectrometry
ICP-MS	Inductively Coupled Plasma Mass Spectrometry
IR	Infra-Red
JAXA	Japan Aerospace Exploration Agency
JSC	Johnson Space Center
LC-MS	Liquid Chromatography–Mass Spectrometry
MNHN	Museum National d'Histoire Naturelle
MS	Mass Spectrometry
NASA	National Aeronautics and Space Administration
OSIRIS-REX	Origins-Spectral Interpretation-Resource Identification-Security-Regolith Explorer
PE	preliminary examination
PMSCF	Planetary Material Sample Curation Facility
PTS	Polished Thin section
SEC	Sample Early Characterization
SEM	Scanning Electron Microscope
SIMS	Secondary Ion Mass Spectrometry
SNOM	Scanning Near field Optical Microscope
TEM	Transmission Electron Microscope
TOF-SIMS	Time-of-Flight Secondary Ion Mass Spectrometry
UV	Ultraviolet
WP	Work Package