EURO-CARES

A PLAN FOR EUROPEAN CURATION OF RETURNED EXTRATERRESTRIAL MATERIALS

WORK PACKAGE 4

INSTRUMENTS & METHODS

PRELIMINARY REQUIREMENTS

(DELIVERABLE D1.4)

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1- Objectives

"The objective of this work package is to establish the most appropriate chain of analyses to perform within the ESCF whilst maximising preservation of the samples and minimising contamination for efficient distribution of samples to the scientific community."

Our aim is to provide a roadmap general enough to establish which sample handling practices and instrumentation are necessary within the facility, which ones would constitute a benefit and which ones do not seem necessary. We notably seek to determine if destructive analyses are required and to assess the risks and benefits of such analyses within the facility. A key issue will be to assess the degree of investigation that constitutes preliminary characterization (i.e. within the facility) vs. scientific analyses (i.e. in laboratories external to the facility). The level, and types, of investigation required for different types of sample return mission also needs to be assessed.

These recommendations need to be flexible as we do not know at present (1) the exact nature and provenance of the sample to be characterized, (2) the scientific goals of the missions returning the samples and (3) the state of the art of instrumentation and methodologies when the samples will be returned to the facility. We restrict ourselves to the cases relevant of EURO-CARES (Moon, Mars and asteroids), in light of the current practices and today's instrumentation but it must be kept in mind that future instrumentation advances may modify the present recommendations.

The present report establishes a preliminary list of sample handling tasks and instrumentation and investigates whether gaps exist between existing or planned instrumentation & the requirements of the ESCF as envisioned today. Current practices in existing sample return facilities (NASA and JAXA) are reviewed as well as those in curation laboratories for extraterrestrial samples collected on Earth. Extensive expert input from the cosmochemistry community and the broader analytical sciences communities is required for this task. The presented tasks and instruments already benefit from such an input, although more will come by further discussing the points listed here. Specific issues relevant of EURO-CARES are (1) instrumentation and methods relevant to life detection and biosafety and (2) contamination control. Assessment of biohazard and bio-burden is specifically relevant of WP2 (Planetary Protection) and will not be discussed in detail here. A summary of interactions between WP4 and WP2 is presented, which will be discussed in detail by WP2 in their D2.2. Finally, a first view of anticipated developments and future instruments is given.

Different samples and different sample curation protocols are to be expected depending on their provenance. Physically, four or five types of sample have been identified: gases, liquids, cryogenic samples and ices, dust particles and rocks. Given that specific sampling protocols are still to be developed for gases and liquids, and that these will likely depend heavily on the specific space mission and given that all present-day facilities curate solid material, we focus this first report on dust and rocks, keeping in mind the curation of cryogenic rocky material. A distinction between rocks and dust may be made at around 1 mm, as is commonly done for meteorites vs. micrometeorites. Such a distinction will impact the sample manipulation protocols. Still it can be kept in mind that hand manipulation of samples remain relatively feasible for grains with diameters down to 100-200 µm with minimal equipment (e.g. tweezers) and that 5-10 µm particles can be handled routinely with sufficient practice and a good micromanipulator.

Samples can also be classified following their provenance. In that respect, the curation methods used in the facility will be different for samples classified as Category V un-restricted according to COSPAR planetary protection guidance, such as those from asteroids and comets, compared to those classified as Category V restricted where they may contain living organisms or hosted life at some time of their history such as samples from Mars or the Galilean satellites. Further difference in sample handling, characterization and bio-burden evaluation will exist if sample comes from known bodies, with already existing returned samples such as the Moon vs. unknown bodies. As mentioned, the EURO-CARES project specifically focuses on Mars, Moon and asteroid samples.

2- Task 1: sample handling

Our most important task is to identify the different actions that will be performed within the ESCF. At present we have identified **eight actions**. Specific problems and questions relative to these actions have also been identified and listed.

i- Sample transport/handling including vialing/de-vialing.

Any sample handling and preparation needs to determine what tools to use and notably (1) the material they are made of, (2) the extent to which they are clean(ed) to avoid contamination and to identify *a posteriori* potential sources of contamination if detected.

An important point is to keep the sample handling as minimal as possible to avoid contamination and loss. A notable issue is transferring the sample from vial to vial. The number of vials and vial transfers for a given sample must be kept minimal. Similarly, vial to instrument transfer for characterisation may also be considered, that will depend on the levels of robotic vs manual handling, sample environment and contamination requirements that may have implications for design of both sample storage containers and instrument interfaces.

ii- Sample characterization

Several questions arose that are related with sample characterization.

The most important issue, already mentioned in the initial proposal is how far the sample characterization must go in the initial characterization phase and to which extent the ESCF must be equipped with the corresponding instruments

An important consideration regarding preservation of sample integrity is to determine how much and what type of investigations can be made whilst the sample is within the space-returned canister compared to removing it from the canister. Keeping the sample in the canister minimises sample handling and protects it from exposure to additional sources of contamination. However, such an approach places restrictions on the types of analyses that can be performed.

An important issue with sample characterization is the extent of sample modifications due to various analytical techniques. Such sample modifications include for instance electron-induced deposition (e.g. carbon-based molecules derived from organics in the SEM), radiation damage (e.g. electron beam damage or UV laser radiation during Raman analysis where the power must be kept below 0.2 mW to avoid structural modifications in organic matter, Quirico et al. 2008), or sample heating by energy deposition.

This latter issue also applies to the sample preparation (point v).

iii- Sample cataloguing

Sample cataloguing is important in order to keep track of all samples at all stages, including those preserved for future generations and those outside the curation facility that have been allocated to researchers. Stages where documentation of the samples will be required include:

- initially returned samples
- pristine untouched samples kept in storage
- characterized samples
- samples allocated
- sub-samples including those complementary to allocated samples
- samples returned from allocation

With several Natural History Museums involved, the EURO-CARES consortium has a long experience of sample cataloguing and allocation. But as far as space missions are involved, the lessons learned from the Apollo samples after 40 years of curation can be extremely useful (2,196 initial individual rocks representing 381.69 kg, now split into approximately 140,000 subsamples, Allen et al. 2011). All handling and analyses performed on the samples must be recorded.

iv- Sample sub-sampling

Sub-sampling can be envisioned for both internal purposes (characterization and curation) as well as to meet scientific requests and optimise allocation of material.

Sub-sampling has the advantage to allow allocation of pristine untouched samples associated with characterized samples, to combine analytical approaches including destructive analyses and to save portions of characterized/allocated samples for future analyses, including analyses not yet developed at the time of allocation. The downside of fragmenting samples is that some information may be lost, such as geometrical relationships between sub-components. After sample call, sub-sampling can be envisioned if scientists request specific sub-components in order to save and store the complementary parts to that allocated. This process may require extensive and careful documentation as reconstruction of samples once fragmented may not always be possible.

v- Sample preparation (e.g. polishing? microtome, FIB, etc?)

The extent of sample preparation will depend on the type of analysis to be performed on the samples. Some specific preparations may be required for certain instruments (e.g. preparation flat/polished surfaces) or sample allocations. A key question is therefore to what extent sample preparation must be done at the curation facility. All sample preparation methods are destructive to some extent. They can be highly specific. Some sample modifications are to be expected during preparation (e.g. fracturing of brittle minerals during microtomy, heavy metals contamination in FIB...). In order to ensure high levels of sample preparation quality and success, and to minimise sample usage it is assumed that most sample preparation will be performed at the curation facility.

vi- Assessment of bio-burden of samples

In the case of Category V restricted samples, determining if life or traces of life are present in the samples and to what extent there are biohazard issues will require highly specific analyses and procedures. The requirements and demands of the sample for these critical measurements will likely be considerable, involving consuming significant amounts of sample for destructive and/or contaminating measurements. This process will also likely be contradictory with rapid characterization and lead to delays in sample allocation to the community. It will also likely require additional complex instrumentation within the curation facility.

vii- Contamination control/knowledge

The environment within the curation facility will be designed to minimise the addition of terrestrial contamination to the samples. This is likely to require complex, high specification systems controlling the air/gases the samples are exposed to, as well as particulate matter, volatile organics, and biological organisms. Monitoring these environments and control systems may require highly specific analyses involving trace gas analysers, particle monitors and a range of bio-burden assessment techniques/instruments. In addition, sample handling/processing is potentially a particularly hazardous time for the samples in terms of exposure to contaminants, and therefore these phases require careful monitoring, through the use of clean analogue samples and witness plates. Such analogues and witness plates need to be defined in interaction with WP5 (Analogues).

Contamination control will be a strong driver for the requirements placed on many aspects of the curation facility, including all materials used in storage and handling areas, instrumentation and sample transfer/transport. Material requirements are to be considered by WP3 (Infrastructure), while it is WP4's role to ensure proper analytical procedure for control. An assessment of the instrumentation required for contamination control will be made, with a careful assessment of whether any contamination-critical scientific measurements of the samples can also be performed with this instrumentation.

Different storage and/or handling conditions may be required to avoid contamination of the samples (e.g. storage or manipulation under inert gases vs. vacuum, as inert gases may introduce contamination for noble gas analyses, Yada et al. 2013).

While all contamination cannot be excluded, knowledge of the nature of possible contaminants will be required by the scientific community analysing the samples. In addition, sub-samples kept under

different conditions could also be considered for keeping some parts pristine for some analyses and some for different analyses. Therefore, as well as curating materials from the spacecraft and the curation facility, witness plates documenting the contamination the samples are exposed to within the curation facility will also need to be collected.

viii- Sample storage

Several issues are related with sample storage that include:

- Evaluation of the steps that potentially exist between sample sitting in curation storage and successful analysis in instrument. What are /must be these steps? To what extent are they potentially damaging to the samples? What sample handling is required to move samples from curation storage to instruments? Robotic handling in curation storage area is a potential solution to minimize the extent of contamination and maximize the output of the curation facility. It is necessary to determine which steps can be automated and what is the amount of development required.
- It is necessary to maintain sample integrity during long-term storage. In that respect the lessons learned from the Apollo samples are essential (e.g. change of grain size distributions, Cooper et al. 2015)
- Long-term storage may require a different specific site. It will be necessary to distinguish storage of pristine untouched samples, from storage of characterized samples, of sub-samples complementary from allocated samples and from samples returned from allocation.

In order to guide the selection of instrumentation required for the curation facility, a database of the information required, including types of measurements, specification (e.g. spatial resolution, area, spectral resolution, elements measured, detection limits, etc) is required.

3- Task 2 : instrumentation

The instrumentation required to undertake the measurements in the ESCF can be further broken down into a number of categories:

(i) characterization of samples

(ii) contamination control/knowledge

(ii) bio-burden/hazard assessment

It is necessary to assess, which methods are required for each of these key activities, and where/if overlap exists that would permit savings in complexity/cost of ESCF.

The types of instruments identified so far for these three activities fall into 5 main categories, which can be strictly non-destructive, minimally destructive or destructive. The impact of each analytical tool on different types of sample needs to be documented – effects such as heating, contamination, radiation damage, etc. The classification of destructive vs non-destructive needs to be carefully assessed. Here is given a preliminary status of the instrumentation list.

(1) Optical methods for documentation

These methods are strictly non-destructive but may still require destructive sample preparation such as 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. Evaluation of the current methods will yield outputs such as definition, optical resolution, illumination and environment requirements, however significant differences may exist at the time of the facility implementation due to continuing analytical developments. It is anticipated that optical documentation of samples at all stages of processing will be 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

Such instrumentation is required to provide additional information relating to the physical nature of the samples. Again it is mostly non-destructive with the caveat of sample preparation. It is essential for preliminary characterization, as well as cataloguing but also for some specific primary analyses (e.g. X-ray CT scan, Ebel and Rivers 2005, magnetic properties, Gattacceca and Rochette 2004) that could be used for classification (Rochette et al. 2003). Among the properties analyzed by these techniques are the magnetic properties, mass of grains and fragments, density, grain density and porosity and internal structure of fragments at different scales. The outputs of evaluation will include identification of most appropriate tools (e.g. X-ray CT), the spatial and spectral resolution that can be achieved, and the time taken for the measurements required.

Examples*: Balances, x-ray CT, synchrotron CT, Magnetic techniques*

(3) Spectroscopic methods

Spectroscopic methods are possibly the best methods to provide provide the characterisation of the mineralogy and chemical composition/nature of the samples with minimal sample handling and sample preparation. Spectroscopic techniques further allow comparison with space analysis of the sample's parent body surface (e.g. infra-red, IR). They are minimally damaging but power of the incident radiation must be controlled (e.g. Raman spectrosocopy) and some sample preparation is required. Evaluation of these techniques will define spectral ranges, spectral resolution, illumination requirements/limits (wavelengths, power densities) and spatial resolution.

An important area requiring careful assessment will be which measurements can be performed on samples within sample canisters, hence constraining the nature of the canisters and/or the limitations on the data that can be produced.

Examples *: Fourier Transform Infra Red (FTIR) spectroscopy, Raman spectroscopy*

(4) Scanning and electron probe methods

These analytical techniques provide high-resolution information (beyond optical) on morphology, chemistry and structure. These techniques can be minimally damaging. Electron induced damage such as ionization or ballistic amorphisation is limited to the sample surface at the depth sampled by the electrons (up to a few μm for SEMs) but significant sample preparation and handling can be required, including sample contamination when a conductive coating is necessary. An important output of this task are to determine (1) the spatial resolution and analytical capabilities of these techniques and (2) to gain knowledge on the sample modification effects and how they can be minimised.

While such instruments might generally not be used on the most pristine samples, they are necessary for advanced sample preparation and will be critical for the assessment of witness plates used for monitoring particulate matter to identify sources of particles. Alignment of instrument capability with the ability to characterise likely contaminants in the clean rooms will be required.

Examples*: Scanning Electron Microscopy (SEM). Transmission Electron Microscopy (TEM) and associated techniques such as Focused Ion Beam (FIB) for sample preparation are highly specific and can only be considered for specific purpose still to be investigated. Electron Microscopy includes associated interactions/detections such as EDX (energy dispersive x-ray spectroscopy) and possibly CL (cathodoluminescence), or EBSD (electron backscattered diffraction).*

(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. Evaluation of these methods will include definition of the elements and compounds of interest, sensitivity, precision, sample requirements as well as an assessment of sample usefulness post-measurement. As most of these techniques are highly specific and destructive techniques, we expect instrumentation within ESCF and relevant of this category to be restricted to biosafety issues and those associated with contamination control and monitoring.

Examples of instruments required for general sample characterisation and chemical contamination include: *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), X-ray fluorescence (XRF), Atomic absorption spectrometry (AAS), Elemental Analyser (EA). Instrumentation required for bio-burden/bio-hazard assessment will be provided by WP2 but some overlap with the general sample characterisation and contamination control instruments should exist that offer opportunities for rationalisation of overall requirements.*

Transfer of the samples between sample storage and the instrumentation facilities needs to be carefully considered. Factors for consideration include: environment control (inert or pure atmosphere or vacuum?), temperature (or even cryo?) control, suitability/availability of common transfer ports on instruments such that standardisation can be achieved for maximum flexibility, contamination or sample disruption risks. For unsterilized Category V restricted samples the level of bio-containment of any sample canisters that may be temporarily removed to instruments for in-canister investigation needs to be carefully assessed. It is anticipated that input from WP2 and WP6 will contribute to this part of the work.

A database of the potential instruments required for the curation facility is required to assess the information that can be provided from each instrument, the sample preparation requirements, known contamination or sample modification risks, sample introduction interface capabilities/possibilities, etc.

In order to ultimately identify which measurements need to be/should be/can be performed in the ESCF a number of additional important pieces of information are required, such as:

- Service provision of each instrument (and compatibility of clean room operation),
- Staff requirements, including level of expertise
- Approximate capital costs
- Approximate annual running costs
- How readily available such facilities are outside the ESCF (but keeping in mind biohazard)
- Number/frequency of measurements likely required by each instrument
- Timescales upon which results from specific measurement types required

4- EURO-CARES improvements compared to existing facilities

4.1 Biosafety issues

All extraterrestrial samples returned to the Earth to date have come from airless bodies unprotected against sterilizing cosmic rays and therefore considered devoid of any traces of indigenous life. This notably includes samples from small bodies for which absence of life was also expected from the study of meteorites and lunar samples, which were shown to be devoid of life after an initial period of quarantine. As a result, bio-burden has not been a major factor in most extraterrestrial sample curation facilities to date. However this is a major concern for future sample return missions, notably for the return of samples from planetary bodies protected by an atmosphere and/or a magnetic field and where habitable zones are postulated to exist.

Taking into account biosafety issues during sample curation is closely linked to the implementation of adequate instrumentation for (1) identification of life or traces of life, (2) planetary protection and (3) implications for sample curation for other purposes.

The definition of instrumentation requirements relevant of life detection and biohazard evaluation will thus face two different challenges:

- identification of usual analytical practices in bio-containment, a first task in itself
- integration of biosafety procedures with sample handling requirements, which we anticipate

is a major challenge.

The study of instrumentation requirements relevant of biosafety is mostly conducted by WP2 (Planetary Protection) in interaction with WP4. As a result, only a brief summary of these interactions and of the strategy proposed by WP2 is given here. More details will be found in the deliverables of WP2. However, an indication of the number and complexity of instruments required for this work has been assessed by Kminek et al (2014):

Solid sample analysis	Gas sample analysis	Liquid sample analysis
• 3D X-ray micro-tomography • Surface imaging and spectroscopy	Not applicable	Not applicable
• Microscopy • Fluorescence • IR, visible, UV, deep UV spectroscopy • SEM	• IR, visible, UV, deep UV spectroscopy	• Microscopy • Fluorescence • IR, visible, UV, deep UV spectroscopy
• SEM, TEM, nano-X-ray-tomography • XRD, XANES • GC-MS, GC-IRMS, FTICR-MS, LC-MS, TOF-SIMS, Nano-SIMS • Target independent biopolymer sequencing	• GC-MS, GC-IRMS, FTICR-MS, LC-MS	• GC-MS, GC-IRMS, FTICR-MS, LC-MS, TOF-SIMS, Nano-SIMS, • Target independent biopolymer sequencing, flow cytometry
Non-destructive and non-invasive	Non-destructive and minimal invasive (no sample preparation	Destructive (specific sample preparation)

Table 1: List of instrumentation required for life detection, bio-hazard assessment and sample characterisation of returned samples from Mars in a workshop report by Kminek et al (2014).

The need to identify suitable microbiology and clean room facilities to understand instrumentation currently used was anticipated. This notably includes facilities that have developed highly specific instrumentation within containment, such as that done at the Fort Detrick facility (MD, USA, de Kok-Mercado et al. 2011) a potentially important contact for EURO-CARES. Other contacts have been developed by WP2. At present the strategy followed by WP2 is a suite of protected environments such as gloveboxes, with decreasing containment requirements, in some ways similar to moveable equipements used for the study of diseases in countries lacking the appropriate medical infrastructure (Allen et al. 2011). In the highest containment parts, instruments will be connected to the glovebox so that samples can be introduced in the instruments without leaving containment. Only the head of the instruments will be within containment, while the major body will be reachable outside containment. Once the search for bio-burden is done, samples are expected to be transferred to a lower containment glovebox if proved or considered to be secure.

A key question is to determine if current instrumentation capability in microbiology and clean room facilities usable for life detection is appropriate for an extra-terrestrial sample curation facility. Several factors will be important, including assessing the impact of microbiology techniques on the substrate (i.e. sample) and amounts of material/surface required. An example of contradictory needs is the air pressure in the clean room : curation facilities commonly use positive pressure for sample integrity (i.e. inside pressure higher than outside pressure to prevent outside dust to enter and contaminate the sample), whereas bio-hazard microbiology facilities use negative pressure for biosafety (i.e. inside pressure lower than outside pressure to prevent possibly contaminated air to escape). To solve such issues and to avoid redundancy in the instrumentation, close interactions will be maintained between WP2 and WP4.

4.2. Contamination issues

One of the primary goals of the curation facility is to maintain the samples in a pristine condition – which includes preventing addition of terrestrial contamination that will affect the ability of researchers to extract the information contained within the samples. In practice, it is impossible to eliminate all contamination, as the very environment and the curation facility itself can and does contribute to material that can interact and add to the samples. In order to minimise this contribution high quality clean room conditions and specially selected materials are normally used. However, monitoring the environment is required in order to ensure that conditions are maintained. Instruments

required will include gas monitoring (or residual gas if samples stored in vacuum), particle counters, residual magnetic fields, and volatile organic content. Bio-burden monitoring may also be beneficial, and essential for samples where life detection scientific goals are set for a specific mission (i.e. restricted Category V samples). Requirements from WP3 (Facilities and Infrastructure) will also provide requirements for the analytical instrumentation required to monitor performance of clean room and other facilities.

Witness plates and sample collection devices will be required to develop contamination knowledge (i.e. to provide information to those analysing the samples) and to allow investigation of any changes to the clean room environment. Instruments that could contribute to this could include analytical SEM, GC-MS, TOF-SIMS, ICPMS as well as a range of bio-burden assessment instruments (from WP2). Cleaning of the clean room surfaces, sample handling tools, sample containers, etc all needs to be monitored to ensure that contamination levels are maintained below specified levels.

Contamination introduced during sample storage, sample transfers, characterisation with the various instruments and sample preparation procedures should be monitored. Optimisation of each of these processes and determining and monitoring the level of contamination associated with them will require use of a range of witness plates and sample analogues identified as part of WP5. A wide range of instrumentation will be required for the optimisation of these processes. It will be necessary to determine which analyses require instruments to be based within the curation facility (on the basis of timeliness or frequency of measurement) and which can be performed at existing commercial or academic off-site facilities.

5- Lessons learned from existing facilities

A number of the questions and points have already been addressed in existing and planned curation facilities. Much can be learnt from these facilities, which instrumentation and methodologies can be considered as a starting point for EURO-CARES. Here we review sample curation practices in the NASA curation facility at the Johnson Space Center, Houston, TX, USA and at the JAXA curation facility in Sagamihara, Kanagawa, Japan in light of the question that are listed above.

5.1. Sample curation at NASA JSC facilities

Much has to be learned from sample curation at NASA JSC. Indeed the strength of the NASA facility is (1) experience with multiple types of samples and associated sample handling procedures, characterization and equipment and (2) experience in long term storage. Only a brief review is given here as a dedicated visit of the curation facilities is planned which will constitute a dedicated deliverable (by WP3).

Five types of sample are currently curated at JSC : (1) lunar rocks from the Apollo missions, (2) rocks from the Antarctic Meteorites collection, (3) interplanetary dust collected in the stratosphere, (4) dust embedded in aerogel collected in the coma of comet Wild 2 by the Stardust mission and (5) solar wind atoms implanted in the collectors of the Genesis mission. Each of these collections has its specificities. A synthetic overview of common practices to these samples and differences is shown here.

All samples are curated in clean room laboratories, with however different levels of cleanliness: class 1000 cleanrooms (ISO 6) are used for the Apollo and Antarctic meteorites laboratories where rocks are handled, whereas the dust laboratories (cosmic dust and Stardust laboratories) are class 100 cleanrooms (ISO 5). The Genesis samples are handled in the highest quality environment, a class 10 cleanroom (ISO 4).

All laboratories use storage in high purity N_2 with positive pressure. Additional glovebox /cabinets with high purity N_2 atmosphere and positive pressure are used for the most precious rock samples (Apollo samples, carbonaceous chondrites and martian meteorites from Antarctica). Other meteorites from Antarctica are cured in air in laminar flow benches. Note that the least precious meteorites are stored in open air cabinets in nylon and teflon bags due to the large number or samples (> 18 000 as of 2011, Allen et al. 2011).

For all samples, hand tools are used. Rules include: minimal contact with the samples and minimal

amount of materials used (only a few types of stainless steel, teflon and nylon). Specific tools are (1) a bandsaw with no fluid (Apollo samples and Antarctic meteorites), (2) a thin section laboratory (same) and (3) a micromanipulation lab (Cosmic dust and Stardust), with the specific equipment to extract and prepare aerogel fragments with embedded dust (called keystones) in the Stardust laboratory.

A common feature of all JSC curation laboratories is the minimal instrumentation used for characterising the samples. It is basically limited to optical microscopy for characterization, weighing (Lunar samples and meteorites) and scanning electron microscopy with EDX for the smallest particles (cosmic dust).

Sub-sampling is systematically used when possible. As mentioned above the Apollo samples have been split into about 140,000 subsamples. Antarctic meteorites are also split and a portion of each sample is sent to the Smithsonian Institution in Washington for the initial characterization of the sample following regular practices in Museums. Note that most of the cosmic dust collected in the stratosphere remained in the collectors.

Finally NASA uses also offsite storage to secure the lunar sample collection in the event of damage of the JSC facility at the NASA White Sands facility in New Mexico. Up to 80% of the Apollo samples are stored pristine and untouched either at the White Sands or at the JSC facility. A portion of the Genesis collectors are also stored at White Sands.

A final lesson from the long NASA experience is that the curation facility must "be prepared for the unexpected". A number of bad events occurred that required specific sample handling and cleaning such as the crash of the Genesis capsule upon return, or the contamination of Stardust by degassing polyethylene...

5.2. Sample curation at JAXA

The Planetary Material Sample Curation Facility of JAXA (Japanese Space Agency) was established in Sagamihara, Kanagawa, Japan, to curate planetary material samples returned from the asteroid Itokawa by the spacecraft Hayabusa. The Hayabusa spacecraft had accomplished a round-trip flight to asteroid 25143 Itokawa and returned its reentry capsule to the Earth in June 2010. It should be noted that the Hayabusa sample curation was planned before its launch with a specific committee dedicated to this task in 2006. Detailed specifications of the facility and the definition of the clean chambers and instruments were considered in parallel. The Hayabusa facility was operational in March 2008, two years before the recovery of the samples from Itokawa (Fig. 1, Fujimura et al. 2011; Yada et al. 2013). The curation facility consists of four rooms with different clean levels: a planetary sample handling room (class 100-1,000), a sample preparation room (class 1,000), an electron microscope room (class 1,000), and a manufacturing and cleaning room (class 10,0000). In the sample handling room, two clean chambers most made of stainless steel 304 were prepared. They are equipped with vacuum systems, pure nitrogen supply systems, electron microscope, cleaning tools and containers, and electrostatically controlled micromanipulation system. In the facility, a Fourier Transform Infra-Red (FTIR) microscope and a dynamic contact angle meter were used to check cleanliness of the surfaces and two mass spectrometers (quadrupole MS and atmospheric pressure ionization MS) were used to check gas purity.

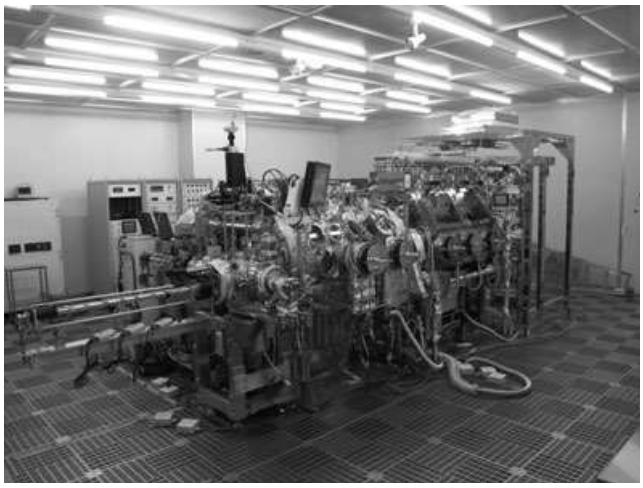

Fig. 1: Picture of the clean chambers in the JAXA Haybusa facility. They are composed of two main chambers located in a room with clean class level of 100-1000.

5.3. Other sample curations

In addition to the curation facilities of the NASA and JAXA space agencies, several other curation facilities led by Museums, Academic Laboratories or Universities have been identified such as meteorite collections (e.g. EURO-CARES Museums, american Museums, the cryo-laboratory at University of Alberta, Canada, designed for curation of the Tagish Lake meteorite), micrometeorite collections (e.g. CSNSM and CEREGE in France, Imperial College in UK). A spreadsheet has been designed to collect information at the level required by EURO-CARES for all such curation facilities. Interactions with scientists leading these facilities are now planned.

6- Future developments and next generation of instruments

An important point to be explored in WP 4 is to what extent instrumentation would be required / can be anticipated beyond that currently employed in curation. This includes (1) the next generation of instruments already available or forthcoming, that will be available and in use at the time of first use of the ESCF, (2) instruments not existing that we may identify as essential and for which partnership with industrial manufacturers should be developed and (3) development of new sample handling protocols /automation.

Such instrumentation developments may be required for non-rocky samples (gases, liquids, icy or cryogenic samples). They may be searched for in other fields of analysis of terrestrial samples – possibilities include glaciology (e.g. Antarctic Lake sample core curation), atmospheric sampling projects (e.g. stratosphere and beyond)

As of today we identified the following promising developments in curation/sample handling on one hand and instrumentation on the other hand:

a. Curation / sample handling

- Cryogenic laboratories installed at University of Alberta and under development at NASA JSC

- An automated robotic arm is under development at NASA JSC. Automated analysis and sample handling chains should be investigated within containment as well as for unrestricted areas, since different constraints are likely in both environments.

b. Instruments

- Cryostage Secondary Electron Microscopes are already available and may be an important component of the ESCF.

- 3D Infrared microtomography and X-ray fluorescence microtomography are under development

and may become available in commercial instruments / without the need for a synchrotron light source in the near future. A related area that may require further development is to address the need for mounting samples without use of contaminating materials (e.g. resins). This could involve use of specialised sample mounts (may be challenging for fragile materials) or static positioning of samples.

- Recent development in electron microscopy resulted in commercial "portable" SEM with minimal size and minimal tuning, which may be useful as part of an automated chain of analysis.

- High precision sample preparation systems are now on the market (tailored for biological and some industrial applications). Similarly, sample transfer devices have been developed by some instrument manufacturers that permit movement of samples from sample preparation/storage to instrument in controlled environments. However, these systems are generally limited to certain sample types and currently only work with certain models within an individual manufacturers portfolio of instruments. An important area for development is to explore how such technology can be utilised for a wider range of sample types and instruments.

A possible way to promote and identify possibilities in automation, simplification and miniaturization of complex instruments, notably to introduce in containment is to stimulate interactions with space mission manufacturers.

7. Conclusions: baselines and key issues

In this document the state-of-the-art in extra-terrestrial sample handling and characterization has been outlined. We have identified current practices and presented key issues. We have tried to isolate the specificities of the EUROCARES requirements. We have prepared the way for the next deliverables of WP4 by identifying the characteristics of the present facilities in the other space agencies. We have clearly identified most of the challenges we will have to face in establishing the requirements for a facility in terms of instruments and methods. Interestingly, one of the key questions will be defining the extent of science that should be performed in the facility. While it is tempting to perform as much as possible, we anticipate that it is more practical and affordable to identify the minimum amount of science that needs to be done within the facility. However, one important case that would be an exception to to this rule is should living organisms be discovered in the facility, a lot of science would need to be performed within the bio-containment zone as samples will presumably not be taken outside the facility. This is however a case whose likelihood is extremely small. In anticipation of that case, the possibility to have instruments move in to the facility, or that time and resource is provided to create a new facility to cater for such a scenario seems at present to be the best option.

Though much of what we build will be implemented from existing facilities and practices (devised by space agencies, natural history museums and curation labs), a few aspects are relatively specific to EUROCARES. Assessing biohazards and contamination issues is not fully addressed by current facilities. The biohazard assessment will be examined by WP2, which will provide the list of required instrumentation. Finally, it needs to be decided how much contamination assessment is required and how much should be performed inside and outside the facility. A base level of contamination monitoring will be defined by standard clean room operations, but additional requirements may be anticipated that are set be each different set of samples and space mission and the specific science objectives of that mission.

Another important task will be to assess how generic should be the proposed facility. At present we do not know what celestial bodies will be sampled, or what physical state they will have, or the amount of samples. So we should keep the planned sample handling and instrumentation as generic as possible without falling into the pitfall of proliferation. The key word should be versatility and minimal requirements for assessing the facility goals: samples characterization in view of rapid and informed distribution, biohazard and contamination assessments.

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