WP1 KNOWLEDGE CAPTURE

PRELIMINARY REQUIREMENTS

THE EUROCARES TEAM

Authors:

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Introduction

This report reflects the data gathered as part of Work Package 1 of the EURO-CARES project: Knowledge Capture and Requirements Review. The report is split into topics that reflect the main work packages of the EURO-CARES project: Planetary Protection (WP2), Facilities and Infrastructure (WP3), Methods and Instruments (WP4), Analogue Materials (WP5) and Portable Receiving Technologies (WP6).

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Work Package 2: PLANETARY PROTECTION

1. Introduction

In this study a review of technology and science requirements in Planetary Protection and contamination control is conducted. Literature publications dealing with Planetary Protection issues for a curation facility's needs for future sample return missions are summarised in this report. This document addresses the issues identifying how Planetary Protection impacts on system technologies and scientific measurements with a final aim to prioritise outstanding any technology shortfalls.

This report addresses Planetary Protection specifically in areas related to the design of curation facility for samples returned from Asteroids, Comets, moons and Mars. Specifically, the major needs are related to:

- Sample containment;
- Transportation;
- Receiving facility;
- Sample handling;
- Preliminary examination;
- Sample extraction;
- Biohazard testing;
- Sample storage;
- Sample delivery to laboratories.

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2. Planetary Protection Overview

Samples returned from asteroids, moon and Mars are subject to satisfy planetary protection regulation. The legal aspects of planetary protection are agreed by all space agencies and they are the guiding principle in the design of space mission to protect solar system bodies from contamination by Earth life forms, and protecting Earth from possible life forms that may be returned from other solar system bodies. The Committee on Space Research (COSPAR) has the mandate from the United Nations to maintain and promulgate the planetary protection policy, both as an international standard on procedures to avoid organic-constituent and biological contamination in space exploration, and to provide accepted guidelines to guide compliance with the wording of UN Space Treaty and other relevant international agreements.

Planetary protection is essential to preserve our ability to study astrobiologically interesting planets and moons of our Solar System. The contamination of celestial bodies by Earth organisms and organics needs to be avoided because it could lead to false-positive results. This would reduce any possibility of detecting native life on that body, if it exists, and a false positive result would also place unnecessarily stringent planetary protection requirements on future missions where these would be in place under the false assumption that life had been found (forward contamination prevention). The second aspect of planetary protection aims to protect the Earth's biosphere from extra-terrestrial agents, which might be harmful if released into the Earth environment (backward planetary protection prevention).

COSPAR defined five planetary protection categories with subcategories dependent on the target of the mission and the type of mission (fly-by, orbiter or lander). Category I missions do not have planetary protection requirements, e.g. for missions to undifferentiated, metamorphosed asteroids or Io. Category V missions include the most stringent planetary protection requirements. All missions which will return extra-terrestrial samples to the Earth for further analysis belong to category V. Dependent on the origin of the extra-terrestrial material a category V mission can be an unrestricted Earth return mission (e.g. with samples from the Moon) or restricted Earth return mission (e.g. with samples from Mars or Europa). For what concerns our work the categories that significantly affect the sample curation solutions for the design and the operational protocols for sample handling are category IV and V. Specifically:

- *Category IV: Lander or probe missions to locations of significant interest for chemical evolution and/or origin of life, and with a significant chance that contamination could compromise investigations (Mars, Europa, Enceladus).*
- *Category V:*
    - Unrestricted: samples from locations judged by scientific opinion to have no indigenous lifeforms. No special requirements (Moon).
    - Restricted: (where scientific opinion is unsure) the requirements include: absolute prohibition of destructive impact upon return, containment of all returned hardware which directly contacted the target body, and containment of any unsterilized sample returned to Earth (Mars, Europa).

As a consequence, future sample return missions from Mars raise serious questions about biological and organic contamination measures that should be taken to protect samples from Earth-sourced biological and organic contamination but also to protect Earth from possible living organism found on the returned samples.

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In this report we will review the state of art of molecular and biological contamination. Techniques for life detection will be showed and, in particular, major studies on curation facility for samples returned from Mars will be addressed.

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3. Contamination issues

Contamination prevention must maintain the samples in their pristine status state for long-term storage (years). Contamination prevention strictly depends on the nature of returned samples. In a first step analysis, the samples returned by missions visiting asteroids, the Moon and Mars will be likely to be heterogeneous regolith with the following principal chemical characteristics:

- *Inorganic compounds:* ferromagnesian silicates, aluminosilicates, Fe and Cr oxides, phosphates, metals, sulphides, carbides, nitrides, and hydrated silicates (e.g. clays).
- *Organic compounds:* soluble carbonaceous and insoluble kerogenous-like compounds, graphite, aliphatic and aromatic hydrocarbons, heterocyclic compounds, amines and amides, alcohols, carbohydrates, biomolecules and, possibly, simple life forms.

It is not excluded that gaseous species and liquids will be also delivered within the re-entry capsule.

Contamination is defined as molecular, liquid and particulate material that could be adsorbed in a certain amount and alter the sample characteristics or degrade the returned samples. Molecular contamination is defined as any gaseous chemical substances. Liquid contamination is any compound (water, organic, metal) with no fixed shape able to flow easily at room temperature and pressure. Particulate contamination is any inorganic, organic and biological tiny (0.01 – 100 mm size) solid particles. Thus, any transfer of chemicals, liquids or particulates of terrestrial origin (environmental, human, processing, facility, equipment and working activity) to the samples has to be minimized.

The contamination prevention must be regularly satisfied during all the following operational tasks inside the Curation facility:

- Earth Re-entry Capsule (ERC) handling;
- Sample handling;
- ERC storage;
- Planetary protection activities (life detection and biohazard assessment);
- Sample storage;
- Sample characterization;
- Sample delivery to external laboratories and retrieving;
- Maintenance of curation facility.

3.1. Contaminants

A partial list of common contaminants that can affect Curation Facility environments are reported in Table 1. Many of these contaminants are generated from four basic sources: the facility, people, tools, and product generated (Brucato et al. 2012).

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All areas adjacent to the cleanroom are generally to be less clean than the cleanroom itself and material airlock and clothing-change area will be contaminated by the activities going on in these areas. It also contains contamination dispersed into it from other sources, such as people, machines and instruments. The floor, walls, ceiling and other surfaces in the cleanroom are examples of surface sources, their contamination being mostly derived from personnel touching them, their contamination depositing from air. Cleanroom clothing, gloves and masks are other surfaces that are contaminated either by the people wearing them or by other cleanroom surfaces. Personnel within the cleanroom can disperse contamination from the skin, mouth and clothing. This contamination can be transferred to the sample through the air, or by contact with their hands or clothing. Machines are another source, as they can generate contamination by the movement of their constituent parts, or by generation by thermal, electrical or other means. Raw materials, sample containers and packaging that are brought in, or piped into the cleanroom, may be contaminated and should be considered as sources.

Table 1 Common environmental contaminants

Source	Contaminant	Type
Facility	Surface coatings: walls, floors and roofs	Particulate & liquids
	Surface desorbed water	Liquids
	Building materials	Particulate
	Air conditioning	Particulate, liquidss
	Room air	Particulate, molecular
	Spills and leaks	Liquid, molecular
	Air filters	Particulate
	Packing	Plasticizers and liquidss
	Containers	Particulate, flakes
People	Skin	Biological (cells), flakes
	Skin fat	Liquids
	Cosmetics	Molecular, particulate
	Spittle	Liquids
	Clothing fibers	Particulate

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	Particles in hair or clothes	Particulate
	Hair	Biological thread
	Bacteria, fungi and viruses	Biological
	Water	Molecular, liquids
	Organics	Molecular
	Secondary microorganism products	Molecular, biofilm
Tools	Friction and wear	Particulate
	Lubricants and emissions	Molecular, liquids
	Vibrations	Particulate, thread
	Brooms and mops	Solids
	Spatters	Liquids, solid film
	Cleaning chemicals	Molecular, liquids
	Plasticizers	Molecular (outgases)
	Adhesive plates	Molecular
	Machine oils	Liquids
Product generated	Teflon	Flakes, molecular
	Quartz	Flakes
	Aluminium	Particulate, molecular
	Gold	Particulate, molecular
	Stainless steel	Particulate, molecular
	Coating metal	Flakes

Known contaminants will be monitored periodically both by passive witness plates mounted in different places of the facility and by active monitoring instruments. Concerning unknown contaminants, tests and analyses should be performed periodically (e.g. outgassing rates as a function of time, chemical composition of outgassing products, condensation rates or degradation as result of radiation). The results of these tests and analyses will be used to calculate expected contamination levels and their subsequent effects on returned sample status if other relevant parameters are known and engineering control need to be developed to remove or minimize to hazardous biological materials.

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4. Mars Sample Containments

Samples returned from Mars can pose a risk to the Earth's biosphere if they contain potentially hazardous agents; therefore breaking the chain of contact between the Martian and Earth environments is a necessity when space mission is designed. For Mars sample return missions (MSR) ESA Planetary Protection Requirements (ESA Planetary Protection Requirements_ESSB-ST-U-001, 2012) requests that the probability that a single unsterilized particle from Mars of ≥ 0.2 mm in diameter is released into the terrestrial biosphere shall be ≤ $1x10^{-6}$. An ESF-ESSC Study Group on MSR Planetary Protection Requirements was asked by ESA to recommend the level of assurance for the exclusion of an unintended release of a potential Mars life form into the Earth's biosphere for a MSR mission taking the newest state of knowledge in the areas of biology, technology, risk assessment and risk perception into consideration. The findings and recommendations of this study were published in 2012 (Ammann et al., 2012). The study group came to the following recommendation: The probability that a single unsterilized particle of 0,01 mm diameter or greater is released into the Earth's environment shall be less than 10^{-6}. The reduced size of the particle within the requirement, 10nm in comparison to ≥20nm, provides a more stringent test for the containment of the facility and its incorporated technologies to conform to the sterilisation limit. This would need to be one of the main factors considered during the design of the facility.

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5. Spacecraft Sterilization

The interpretation of sample analyses once studied on Earth is critical because science-driven organic contamination control is inextricably coupled to Planetary Protection measures. While some techniques to control the chemical cleanliness of surfaces are also effective in reducing the bioburden, others may work against the science goals. An example is the use of alcohols by spacecraft engineers for cleaning surfaces. Ethanol or isopropanol are, in fact, used by biologists to inactivate the majority of bacteria. However, these alcohols cannot be used for sterilization because they are not effective against bacterial endospores and some other robust microorganisms. Alternative techniques for sterilisation and decontamination are used in the microbiological industry, such as gaseous disinfection where a chemical is vaporised into a sealed enclosure and acts on the organisms present on the surfaces. There are a number of gaseous decontamination technologies commercially available, and standards are available for their use (ECSS-Q-ST-70-56C: Vapour phase bioburden reduction for flight hardware). But whilst these techniques will inactivate the microorganisms present on a surface, the remaining whole organism or components of it would still be present on the surface and present as chemical/biological contamination which could lead to issues in interpretation of any test undertaken. Of the many Planetary Protection technology initiatives that will be explored to design the curation facility, the cleaning and contamination control measures are most directly linked to science requirements.

The exposure of spacecraft materials, components, subsystems and also of whole spacecraft, as previously undertaken for the Viking landers, to dry heat microbial reduction (DHMR) in a standardised process was the first bioburden reduction technique officially accepted by space agencies. As defined in the ECSS standard, which is used by ESA and NASA (ECSS-Q-ST-70-57C, August 2013), different combinations of temperature and exposure time have to be used for bioburden reduction depending on the chosen process (humidity controlled or not), the necessary bioburden reduction level, the aim to reduce the bioburden on ambient surfaces or on mated surfaces, or the bioburden encapsulated in spacecraft materials. This has been and continues to be a valuable and practical technique for many types of hardware.

As an alternative, the capability to sterilise the appropriate elements of spacecraft with hydrogen peroxide is being developed (Chung et al. 2008). Recent performance advances in electronics and other thermally sensitive components makes the use of high temperatures unsuitable for some hardware. Experiments to evaluate the effectiveness of vapour hydrogen peroxide (VHP) in a small vacuum chamber system for the inactivation of the standard spore challenge, *Geobacillus stearothermophilus*, were conducted by STERIS Corporation and reported in Chung et al. (2008). Results demonstrated that VHP provided significant reductions in spore viability while allowing survival of sufficient spores for statistically significant enumeration. The study delineated a range of test steriliser process conditions: VHP concentration, process duration, a process temperature range for which the worst case D-value may be imposed, a process humidity range for which the worst case D-value may be imposed, and the dependence on selected spacecraft material substrates. Further work was completed using varying concentrations VHP and chlorine dioxide technologies at ambient pressure in a large chamber (~20m^3) by Pottage et al. (2012). This test was conducted with a range of Bacillus *spp.* isolated from spacecraft assembly clean rooms and studied the inactivation kinetics through the decontamination cycles. The derivation of D-values from the lethality data permitted conservative planetary protection recommendations, which have been incorporated into the ECSS standard ECSS-Q-ST-70-56C (August 2013), which is used by

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ESA and also by NASA summarizes the experimental details for using VHP for bioburden reduction in both vacuum and non-vacuum chambers.

The sample collection and containment tools, once sterilized, will need to be isolated from other parts of the spacecraft. To isolate the components and systems after sterilization, lightweight biobarrier material needs to be developed that can be applied to different sized objects, and a number of materials are currently in the test phase. A biobarrier could also be produced that would encapsulate and entire spacecraft and would then be used to protect it from the launch vehicle. Another key development identified is a technique for collecting clean samples from beneath the Martian regolith surface, which might possibly be contaminated by migration of microbes from a "dirty" lander or rover. An interesting alternate path involves investigation of techniques to chemically tag spacecraft contaminants so that these could be recognized in the sample upon return. The techniques for sterilization of spacecraft subsystems being investigated are heat, VHP, plasma, UV irradiation, and gamma radiation (see e.g. Gersham et al. 2004).

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6. Planetary Protection techniques and protocols

In addition to extensive decontamination and cleaning of the outbound spacecraft, the return samples potentially containing extra-terrestrial life requires a container that can be remotely closed, sealed and monitored to ensure the external surfaces are ensured to be free of external contamination, safely launched from the planet, monitored en-route, and opened in an appropriate Sample Return Facility (Rummel, NASA white paper).

According to MEPAG E2E-iSAG, 2012, three distinct classes of samples should be returned in order to have a high scientific return; namely rocks, grains such as regolith and sand, and atmospheric samples. Thus the techniques necessary to detect possible presence of life in samples and that are needed to be present inside the Sample Curation Facility should be able to analyse solids, liquids and gasses.

6.1. Analytical techniques

To define the techniques that are essential to detect life signs in the samples returned from Mars, a workshop took place at the Scripps Institution of Oceanography, La Jolla, California in February 2012 organized by NASA and ESA on life detection in samples returned from Mars. The workshop was held the days after the Life-Detection Science Conference, which was organized to discuss, to determine and to assess the latest concepts and methods to search for life on Mars samples (Allwood et al. 2013). Many open questions were discussed during the workshop and the main topics were:

- To review the Planetary Protection Draft Test Protocol as reported by Rummel et al. 2002;
- To identify research and technical developments that are necessary to establish and execute a future Planetary Protection Test Protocol;
- To identify major issues that might affect the requirements necessary to design future sample return flight hardware.

A general consensus was achieved among the members of the Working Group in defining a sequence of analyses to be executed both during samples acquisition on the Martian surface and when the samples are inside the Sample Curation Facility. The suggested analyses were defined to ascertain the presence of life in the samples and the techniques proposed should be able to characterize solid, liquid and gaseous species. The proposed sequence of analyses - as published in the workshop report by Kmineck et al. 2014 - are here showed in Table 1.

Table 1. Sequence of sample analysis to be performed on Mars surface during sample collection and on the samples once delivered to Sample Curation Facility as showed by Kmineck et al. (20014).

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Sequence for sample analysis	Sample condition	General type of analysis
I	Sample acquisition on Mars	Remote and *in-situ* analysis on Mars to characterise the sample type and the geological context
II	Any solid sample material on the outside of the sample containers	Solid sample analysis; full sequence (non-destructive & non-invasive, non-destructive & minimal invasive, and destructive)
III	Head space gas	Gas sample analysis; full sequence
IV	Solid samples in containers	Solid sample analysis; non-destructive & non-invasive
V	Solid samples removed from containers	Solid sample analysis; non-destructive & minimal invasive
VI	Fluid inclusions from solid samples removed from containers	Liquid sample analysis; full sequence
VII	Solid sample removed from containers	Solid sample analysis; non-destructive & minimal invasive, destructive

According to the consensus obtained among the participants of the La Jolla workshop, the analytical techniques that were defined as appropriate according to the nature of the samples to detect sign of life on Martian samples were:

Fourier Transform Ion Cyclotron Resonance Mass Spectrometry (FTICR-MS);

1. Gas Chromatography Isotope Ratio Mass Spectrometry (GC-IRMS);
2. Gas Chromatography Mass Spectrometry (GC-MS);
3. Infrared Spectroscopy (IR);
4. Liquid Chromatography Mass Spectrometry (LC-MS);
5. Scanning Electron Microscopy (SEM);
6. Transmission Electron Microscopy (TEM);
7. Time of Flight Secondary Ion Mass Spectrometry (TOF-SIMS);
8. Ultraviolet spectroscopy (UV);
9. X-Ray Absorption Near Edge Spectroscopy (XANES);
10. X-Ray Diffraction (XRD).

As reported in Table 2, the techniques were sorted out according to the nature of the samples. Further considerations were made on the invasiveness of the analyses.

Table 2. Techniques for Life-Detection as defined by the Workshop on life detection in samples from Mars (Kmineck et al. 2014)

Invasiveness	Solid sample analysis	Gas sample analysis	Liquid sample analysis
Non-destructive & non-invasive	• 3D X-ray micro-tomography • Surface imaging and spectroscopy	Not applicable	Not applicable
Non-destructive & minimal invasive (no specific sample preparation)	• Microscopy • Fluorescence • IR, visible, UV, deep UV spectroscopy • SEM	• IR, visible, UV, deep UV spectroscopy	• Microscopy • Fluorescence • IR, visible, UV, deep UV spectroscopy
Destructive (specific sample preparation)	• 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

The detection of any possible life form is based on the paradigm to search of life as we know it. Thus biochemistry that governs all the processes of terrestrial organisms is assumed to be active on Martian organisms. Thus, portion of sample returned will be analysed to detect

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biosignatures starting from a broad survey of different portions of sample types and continuing with deeper high resolution analysis. To this aim we can organize the techniques showed above according to their capability to provide information on morphological, chemical, biochemical, isotopic and mineralogical signatures. Furthermore, we have here evidenced the specificity of each technique to detect multiple independent signatures. The detection and interpretation of these signatures is the way with which the life detection is based. Thus, techniques and capability to detect biosignatures are showed in Table 3.

Table 3. Techniques for life detection as from Kmineck et al. (2014) are here showed according to their capability to detect biosignatures.

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BIOSIGNATURE		TECHNIQUE: Electron microscopy - SEM	Electron microscopy - TEM	GC-MS	LC-MS	IR spectroscopy	Gas Chromatography Isotope Ratio Mass Spectrometry (GC-IRMS)	XRD	UV spectroscopy	XANES	TOF-SIMS	Fourier Transform Ion Cyclotron Resonance Mass Spectrometry (FTICR-MS)
Morphological	Size of single cell - Size of targets	X	X									
	Numbers of single bacteria - Number of targets	X	X									
	Population size (colonies)	X	X									
Chemical	Chemical composition			X	X	X	X			X	X	
	Chirality			X	X		X				X	
	Organic molecules		X			X	X		X			X
Biochemical	DNA, RNA										X	
	Biopolymer (e.g. carotenoids)					X					X	
	Protein										X	
Isotopic analysis	Isotopes, Isotopologues			X		X	X					X
	Isotopomers											X
Mineralogical	Elemental analysis	X										
	Mineral structure							X	X	X		
	Structure, Mineralogy					X		X				

In this project the above list of instruments for life detection is the subject of a review not only by the member of WG 2, that are focused on planetary protection, but also by members of WP4 interested on the characterization of the samples. Implementation is based on the fact that instruments that are identified as essential for Planetary Protection can be also used for preliminary characterization of samples. To this aim we have started to extend the methodologies for life detection defined by Kmineck et al. (2014) in order to encompass the preliminary organic, mineralogy and isotopic characterization necessary to provide the first description of the nature of samples necessary for extended studies that will be performed in worldwide laboratories.

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A more articulated approach to life detection and biohazard methods and protocols are presented in NASA/CP-2002-211842 (2002). NASA draft test protocol was developed considering that important effort has to be made to perform a preliminary analysis of the physical-chemical characteristics of the sample to detect signs of life and execute biohazard testing before the samples are released to the broader community for scientific analysis. Isolation and containment of a potential biohazard requires that direct interactions between humans and the sample must be minimized because the most common causes of containment breaches are associated with human handling. The ideal system would provide protection for both the sample and operators of sample curation facility. Largely or totally autonomous sample handling and analysis capabilities are needed, reducing the parallel risks of inadvertent loss of Mars sample containment and sample contamination.

The approach that was used in NASA draft test protocol for detecting life is based on omni-comprehensive methods that not only can identify the organic and geochemical signatures but also is able to evidence silent or still active metabolism or replicating activity. Biosignatures are, thus, identified and can be summarized as in Table 4.

In order to rule out any terrestrial contamination of the sample standard microbiological examination procedures (e.g. cultivation, amplification techniques such as polymerase chain reaction, sequencing methods) should be applied to aliquot of samples. This implies further complexities in designing dedicated environments inside the sample curation facility that must be of high containment, i.e. as those typically used in BSL 4 facilities. The protocols developed to detect life should be reviewed periodically as part of the ongoing processes that will incorporate new laboratory findings, advances in analytical methods and containment technologies.

Table 4. Biosignatures for life detection (modified from NASA/CP-2002-211842)

BIOSIGNATURE	
Morphological	Size of cell
	Shape of cell
	Structure
	Morphological indication of replicator or specialized features
Structural chemistry	Membrane
	Attachment and motility structures
	Polymer
	Organics or complex molecules
Metabolism and Bioenergetics	
	Products result from specific enzymecatalyzed reactions
Biosyntetic Mechanism	Protein-enzymes
	Protein
	Amino acids
	ribozimes
Isotopic analysis	Isotopes, Isotopologues
	Isotopomers
Geochemical Signatures	Magnetite
	C,H,N,O,P,S in pigments
	Iron, Molybdenum, Tungsten

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6.2. Biohazard

Samples returned from Mars have to be considered carrier of agents that can pose threat to life on Earth. Since potential hazards can be of various nature, e.g. chemicals, antibiotic-resistant bacteria, rapidly-mutating viruses etc., the methods and protocols to carry on in the sample curation facility should be diverse. To prevent any threat to personnel operating inside the facility and to environments, development and refinement of high containment areas in which infectious microorganisms could be safely handled is mandatory. The needs to work with tissue cultures, maintain sterility of cell lines, and minimize cross-contamination contribute to concerns regarding activities plan and facility design. Since the workers are the main source of contaminants (Table 1), samples have to be protected from personnel contamination further than to protect the worker from the risks associated with the manipulation of potentially hazardous materials.

Areas of the facility should be dedicated to whole-organism animal and plant *in vivo* tests and cellular assay and molecular biology *in vitro* tests. Since samples should be considered as dangerous exotic agents that may pose high risk to life-threatening disease, maximum containment equipment (BSL-4) should be used for all procedures and activities. This means to have separate building or isolated zones where biohazard tests will be carried on. Supplies will, thus, be dedicated decontamination system foreseen. However, the ongoing approach is to reduce the use of animals in biohazard testing replacing by cellular and molecular analyses.

The use of proper procedures and equipment cannot be overemphasized in providing primary personnel and environmental protection. The most important piece of containment equipment, however, is the biological isolation cabinet in which robotic and/or human manipulations of hazardous agents are performed. The safety cabinets, within which the maximum level of cleanliness should be maintained, will provide the primary barrier between the sample and the environment. Implementation details discussed in previous studies vary considerably according to number, connectivity, approach to sample handling, and so on (Beaty et al. 2009).

The Draft Test Protocol (Rummel et al 2002) refers to conventional whole organism animal and plant in vivo testing. Thus, a significant part of the floor space of the containment laboratory core should relate to animal holding. Since the Draft Test Protocol was published in 2002 the fields of microbiology and biohazard detection have advanced markedly. To this aim a dedicated workshop is foreseen to be held in Firenze 14th -16th June 2016 addressing the state-of-the-art practices and future implementation. Thus, the design of the sample curation facility would accordingly be simpler.

Three concept studies for infrastructure of a Mars sample receiving facility was investigated and discussed by Beaty et al. (2009) with main aim to define basic attributes. The main activities that were foreseen in such studies are to receive the Earth Re-entry Capsule (ERC)

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directly from the landing site with preliminary check and external surface cleaning, disassemble the ERC extract the sample canister from the ERC and initiate subsampling and physical tests, extract the samples from the canister and initiate the life detection testing, perform biohazard tests on samples and finally store samples in pristine environments. The sequence of actions is summarized in Figure 1.

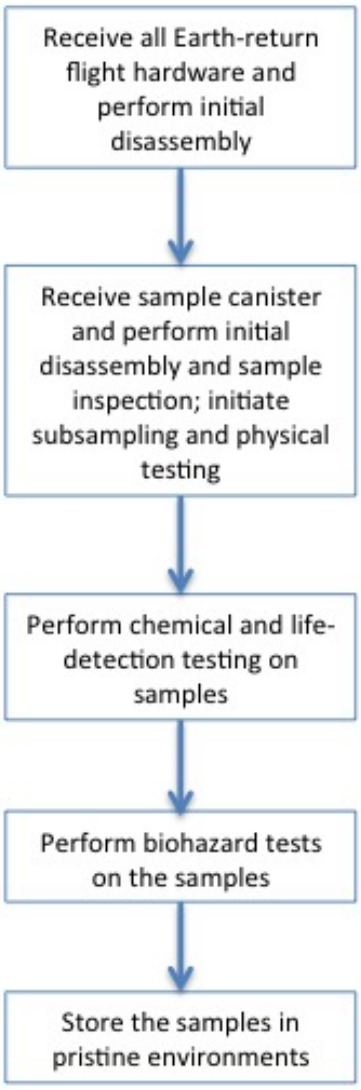

Figure 1. Sequence of actions to be performed in the Sample Curation Facility (modified from Beaty et al. 2009)

The SCF is designed in our view to perform also preliminary characterization analyses of the samples, to catalogue the samples as well as to enwrap the sample containers to send them in to other facilities and worldwide laboratories. Thus, a further activity we envisage to be added that is missed in Beaty et al (2009). After biohazard tests, sample characterization and cataloguing has to be performed before to prepare the samples for their storage.

These requirements lead to the definition of operational and architectural specifications for a sample curation facility that would be handling potentially contaminated returned samples (Mancinelli 2000, Mani et al. 2007, Allen et al. 2011). This facility requires the combination

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of engineering technology and interventions used within high containment microbiological laboratories (BSL-4) to maintain the safety of the workers and environment from the sample being handled, with cleanroom engineering and technology to maintain the sterility of the sample from the Earth environment. This type of containment is stringent, including (but not limited to) such requirements as; having the facility under negative pressure in comparison to the external environment (see Figure 2), all personnel change into protective clothing before entering the facility, entry and exit through air locks, all personnel must shower before exiting, all air leaving the facility is filtered to assure no biological agent is released.

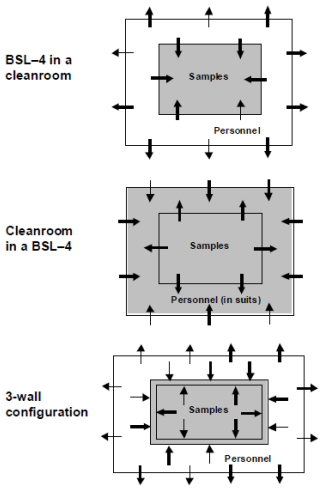

Figure 2. Different options for the combination of a high biological containment facility with a cleanroom design (Rummel et al. 2002).

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7. Conclusion

Conclusions from three conceptual studies summarized by Beaty et al. (2009) stated that most of the technology needed for the design and to construct a Mars SCF already exists within the biosafety, pharmaceutical, and sample curation communities. But some technologies and techniques, such as surface decontamination, double-walled glovebox containment and dexterous ultra-clean robotics will need to be developed. Once adequate records of chain of custody of samples are ensured, the tools and instrumentation would be accommodated inside cabinets or on laboratory benches, depending on the specific approach to the containment barriers and sample manipulation. This is subject of investigation in this project. A further specificity that needs to be developed is the interfaces between life-detection instruments and double-walled environments where the samples are contained. Off-the-shelf instrumentations are designed to be operative in common analytical laboratories thus, a further detailed study how to modify analytical instruments to be accommodated within double or single walled chambers is needed.

Materials by which instrumentation is built (e.g. electronics, plastic, metals, pumping system, gas supplies etc.) are not conforming to ultra clean and sterile environments like those envisaged for sample curation facility. Tools in contact with samples, and the sample chambers where life-detection instruments will be placed would need to be cleaned to the same standards as for the sample-contact surfaces. Standards and protocols to achieve this were not developed yet and, thus, it's a matter of further investigation in this project. Instrumentation for life detection will affect the facility design and, thus, they will need to be known before the construction of the facility.

Methodology for biohazard assessing for the detection of pathogenic organism needs to be reviewed and implemented according to new findings that once validated would replace many current practices. Microbiology and biohazard detection have advanced markedly in last years encouraging in vitro tests instead to conventional use whole organism animal and plant in vivo testing. This will largely simplify the design of the facility. Under this project, a dedicated workshop is planned for accounting on new findings in biohazard assessment (14th - 16th June 2016, Florence Italy). The topics to be discussed will be:

- Techniques and methods of risk assessment of biohazard;
- Test methods for the detection of pathogenic organism;
- Containment systems;
- Sterilization processes;
- Safety equipment, enclosed containers;
- Procedures to monitor the health of personal and select precautions for safe practice.

Planned sampling methodology and sample preservation is matter of further study due to the fact that some chemicals decay on exposure to light, or microbes may induce decomposition of organic compounds at ambient temperatures. Moreover, chemically reduced compounds may oxidize on exposure to a head space of air and volatile compounds may escape from an insufficiently sealed vial.

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Analytical instruments for life detection as defined by Kmineck et al. (2014) will be subject of review. Many techniques could be used both to detect biosignatures and to characterize samples as mineral contents, petrology, isotopic and organic composition. Thus, a collaborative interaction with WP4 will implement current state of art in facility endowment.

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Work Package 3: FACILITIES AND INFRASTRUCTURE

1. Introduction

We summarize here the current knowledge on facilities to receive, contain and curate extra-terrestrial samples while guaranteeing terrestrial planetary protection based on a literature survey. The main requirements and important information identified during our knowledge capture are summarized in this preliminary report.
We have covered in this "knowledge capture and requirements review" all the different aspects of the 3.1 task of the Work Package 3 (WP3), from the building design (construction, operation, security, etc.) to the storage of the samples (monitoring, sample holder, etc.) to the long-term curation of samples (sample handling, database, etc.).
At the moment, this review is mostly based on what is known from published literature on 'equivalent facilities' at the NASA Johnson Space Centre in Houston (USA) and at the Planetary Material Sample Curation Facility (PMSCF) of the Japan Aerospace Exploration Agency (JAXA) in Sagamihara (Japan), knowing that none of these facilities meet all requirements of sample return missions from Mars, i.e. these facilities are not currently capable of handling materials with planetary protection requirements. We also utilised the almost two and half centuries of experience accumulated in curating meteorites, in various laboratories and museums around the world.
We plan, in a second step, to extend the present review with the collection of expertise from planetary scientists and to gather information from biosafety laboratories, cleanroom manufacturers, electronics and pharmaceutical companies, nuclear industry, etc.

The experience from past sample return missions is invaluable in addressing the future challenges of planning and building a 21st century facility, however, scientific (and political) developments should also be taken into account.

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2. Building design

Main requirements: The infrastructure should be designed and constructed both to prevent terrestrial sample contamination and alteration on one hand, and to prevent the release of potential biohazards from the sample on the other hand. All measures should be taken to avoid contamination from terrestrial particles and organisms but also terrestrial gas and liquid contamination, this is especially important for Mars samples where contamination by e.g. organic species and/or terrestrial biology could impair testing for putative past or extant Mars life. In addition to stringent cleanliness requirements, it should also operate under the strict guidelines of the United Nations space treaties on Planetary Protection and others as developed by international and national space and environmental protection/public health agencies. It should also allow the samples to be studied by the international scientific community, either within the facility itself, or in others laboratories, through loan of samples.

The purpose of such a receiving and curation facility is (1) to take delivery of the returned spacecraft, (2) to open the spacecraft, (3) to extract the sealed sample container, (4) to open and to recover the samples (rock, dust, head of gas, etc.) from the sample container, and then (5) to transfer samples to the curation laboratory (i.e. where samples will be curated; for more details see below the "Curation section"). If applicable, depending of the origin of the samples, biohazard and life detection tests are also likely to be conducted within the facility.

For some samples from Category V restricted bodies (e.g. Mars), life detection is a very important topic, not only in term of the "biosafety/planetary protection perspective" (i.e. we need to protect our planet from contamination by any potentially harmful living extraterrestrial organisms that may be contained in the returned samples), but in the "science perspective" (i.e. to know whether life ever arose on Mars). Accordingly, specific and appropriate handling and analysis of these samples is required. Inputs from the WP2 will provide all the necessary requirements for the best appropriate way to deal with these sensitive samples.

Biosafety level 4 containment (BSL-4; the highest level of containment) facilities are designed to contain the most hazardous microbiological agents on Earth and to prevent them from infecting staff or being released to the environment. Most facilities depend on the operators wearing positive pressure suits but a number of facilities use cabinet lines (negative pressure glove boxes) to contain the samples ("agent"). These facilities contain the samples mainly by the use of a number of engineering controls including filtration, negative pressure, sealed facilities, gaseous disinfection and primary containment systems (safety cabinets, isolators and glove boxes). Waste is treated by autoclaving, heat and chemical treatments.

Different parts of the facility: A conceptual design of facility should include several distinct, but connected (i.e. via pass boxes, doors, etc.) adjacent cleanrooms/laboratories (with increasingly positive pressure toward the most pristine areas), namely:
(1) a receiving laboratory,
(2) a containment laboratory (with isolation cabinets as well as a "secondary containment barrier"),
(3) a cleaning (and sterilization) room (equipped with ultrapure water, a CO2 blast cleaner, an UV ozone cleaner to sterilize bacteria and remove organics of containers and tools, etc.),

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(4) an opening laboratory (equipped with (Viton gloves) steel cabinet(s) under conditions of pure, positive pressure nitrogen or other inert gas),
(5) a curation laboratory (with a number of (Viton gloves) steel cabinets) and
(6) a storage (vault) room (with sealed containers).
In addition, a specific room designated to support instrument development and testing can also be incorporated to the facility concept.
Importantly, the general arrangement and connection between these different cleanrooms/laboratories will highly depend on the human versus robotic handling. This important point is also discussed to some extents in the "Curation section", but it is already a key point to take into account in the building design in general. Both approaches, i.e., human versus robotic, have advantages and disadvantages that will have to be further investigated in ongoing work.

More generally, office space for the permanent staff, as well as for guest staff, should be incorporated in the general concept.

Technical requirements: Cleanrooms should be equipped with different vacuum systems (such as turbo molecular pumps and dry scroll pumps), with pure nitrogen supply systems and with pressure control systems (typically positive relative to atmospheric pressure to minimize contamination). One of the main requirements and a problem in combining high-level biological containment with cleanroom conditions is that, to maximize biocontainment, the air pressure should typically be negative relative to atmospheric pressure, when, as mentioned previously, a positive pressure is needed to maximise cleanliness and minimize contamination.
Air-handling systems are a critical environmental controls in such a facility and should be properly designed (if not even duplicated in case of a failure of the main system). Typically the filters used in filter fan units should be made of polytetrafluoroethylene (PTFE) and coupled with a chemical filter.
For the flooring of the cleanrooms, an anti-static floor is more than recommended and rounded corners are best, to avoid build-up of dirt and to aid thorough cleaning. Vibration sources should also be avoided and/or mitigated as vibrations are problematic when manipulating small samples or using sensitive analytical equipment, such as balances for weighing samples.
Sample handling (Viton gloves) steel cabinets, with flow nitrogen gas, should be equipped with air lock(s) to be able to introduce diverse tools, containers, etc. without breaking the environment inside the cabinets. Methods to mitigate electrostatic charge build-up, such as an alpha-ray neutralizer (^{210}Po radioactive source), are also very important.
From a contamination point of view, only a very restricted variety of materials can be introduced inside the cleanrooms and in particular within the cabinets, typically only stainless steel (304 and 316 grade), pure aluminium and specific aluminium alloys, quartz glass and PTFE are allowed. These materials have a low potential of contamination, or have a simple and known composition that can be recognized as contaminants if detected in samples or on witness materials. Materials that possibly off-gas into the cleanrooms and especially within the cabinets should be avoided or carefully controlled.

A waste sterilization system should be planned to be able to sterilize both liquid and solid waste products prior to releasing them in the environment.

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The facility should be equipped with a system allowing communication by both audio and video, to facilitate communication between different laboratories within the facility and also to the wider world with and to external stakeholders, e.g. investigating scientists or other relevant experts. An access to the internal server of the facility should also be available, to be able to update in real time the sample database.

Important environmental systems for controlling the environment of the cleanrooms and associated equipment such as the glove boxes (pumps for the vacuum systems, ultra pure water supply system, nitrogen purifiers, etc.) should be incorporated in the general design of the facility.

Whenever possible transparent apertures should be incorporated in the design of the cleanrooms/laboratories to allow a maximum of the visiting scientists (and if appropriate the general public) to observe the facility without having to physically enter critical parts of the laboratories.

Security and risks management: The entire facility should be designed in a way to ensure a high level of security, with restricted access and tracking systems. In term of safety, a specific fire protection plan and secondary power supply system and other types of backup systems are also mandatory due to the specific operations to be performed under controlled conditions. Depending on the location, additional security measures will be considered (hurricanes, flood, etc.)

In addition, the need of a suitable and secured remote storage of a representative subset (% TBD) of each of the curated extraterrestrial samples will have to be considered to ensure that the entire sample collection is not contaminated, damaged or even lost in the event of an accident. In typical remote storage facilities (e.g. White Sands for NASA, US), samples are kept in “dead mode” under an inert gas atmosphere.

Important issues such as feasibility, cost estimation and timescales are not discussed here. However, it is already clear that the planning of the facility design needs to start early as possible, several years before planned return sample date and that such a facility will have to preserve (and protect) samples for decades so as to allow years of research to be carried out on them.

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3. **Storage of the samples** (monitoring, sample holder, etc.)

The facility will have to operate at controlled pressure, temperature and atmospheric environment (especially the relative humidity). All these parameters should be carefully monitored.
At least three types of sample storage will have to be considered: (1) "unopened storage", for unprocessed samples, (2) "working storage", for processed samples (designated for study and loan to other laboratories, etc.) and (3) "readmitted storage", for samples that have been studied in other laboratories and returned to the facility.
The pristine samples should be preserved from hydration and oxidation in an atmosphere of high-purity nitrogen gas. The nitrogen should be checked for trace contaminants (water vapour, oxygen and noble gases), and the isotopic ratio should be monitored.

The temperature and pressure conditions should be kept as low as possible, to be as close as possible to the conditions that the samples encountered prior to return to Earth. From the sampling on the parent body to the storage in the facility, P, T and other physical parameters should be recorded, with loggers and witness plates. Currently, almost all curated extraterrestrial samples are stored at room temperature (only a very small fraction of samples are preserved at sub-freezing temperatures, for example samples of the Tagish Lake meteorite). Curating frozen samples introduces significant challenges, but the appropriate storage temperature (and environment) will have to be first defined and then maintained over time. Routine curatorial procedures, especially sawing (to a lesser extent splitting and sieving) will have to be adapted to prevent the sample temperature from rising. Shipping procedures that maintain the samples both "cold and clean" will have to be developed, tested and certified (WP6 is investigating these options).

The potential contamination of the samples should ideally be monitored during acquisition and collection during the mission by using witness materials/coupons which could be placed inside the sample catcher/container on the spacecraft. As with the samples themselves, these witness materials should be curated under the same conditions, i.e. P, T, atmosphere and will serve as important samples themselves, especially should any potential sources of contamination from the spacecraft and spacecraft operations be identified.

Another point to be considered is the magnetic properties of the samples. Magnetic properties can be affected directly by the spacecraft propulsion system, and then during the atmospheric re-entry, landing, transportation to the facility and also within the facility. For future missions, ways to protect the sample from potentially deleterious magnetic fields should be encouraged. Once inside the facility, appropriate measures will have to be defined. In every case, the disruption of the sample magnetic properties should be assessed and quantified with the use of analogue materials, and large magnetic field sources should be (as much as possible) banned from the facility.

In term of monitoring, possible alteration of the samples during their stay inside the facility should also be controlled. A thorough understanding of how the curated samples "react" to the storage conditions is mandatory. Even if all necessary measures will be taken to maintain sample integrity within the facility, it cannot be totally excluded that some alteration (of whatever type) of the sample will occur.

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The environment of the cabinets, as well as the surface of tools and materials being in direct contact with the samples will have to be carefully monitored and controlled. The use of specific mass spectrometers could be envisaged for this purpose.

Different types of sample holders and storage containers should be envisaged, depending on the specificities of the samples and subsamples, such as aluminium containers, stainless steel containers, or precision-cleaned quartz-glass containers (knowing that the different types of listed materials can also be combined via surface coating). For long term storage, samples should be packed under nitrogen gas and sealed in multiple Teflon bags themselves sealed in gas-tight aluminium and stainless steel cans. Alternative types of sample holders and storage containers will have to be considered and designed based on the specificities of the samples to be curated and on the specific requirements from the scientific community and from inputs from the WP4. When applicable, the sample return containers themselves can be used for storage, especially for pristine samples. For long term storage all the sealed containers should be secured in locked nitrogen steel boxes, or cabinets maintained with positive pressure with respect to the room, under a constant flow of nitrogen gas within a high-security storage vault.

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4. **Curation** (sample handling and database)

Curation of extraterrestrial samples is a very critical step at the interface between sample return missions and the research community. Curation mainly consists in the collection, handling, documentation, preparation, preservation ("into the indefinite future") and distribution of a limited amount of sample for research. Education and public outreach is also part of the duty of curators; however, this specific aspect is not further developed here.
Curation should already start with mission design. All steps, from the collection of the samples until the arrival in the facility, should be properly documented. Curator(s) should already be consulted during the mission design, not only as expert of the samples to be collected, but also to be able to help in the design of the sampling device(s) (to be attached on the spacecraft) and to ensure proper monitoring of the contamination (already during the construction of the sampling device(s)).

High adaptability: Importantly, each of the collected and curated samples has a unique (and distinct) history and comes from different environments. Consequently, the different types of samples present specific and unique challenges with respect to curation. Therefore, the curation of these samples should follow strict and sufficient procedures to insure their integrity. These procedures will have to be adapted to the samples to be returned from future space missions (i.e. taking into account of the diversity and special requirements of the samples) and also updated according to the evolving needs of the research community.
Samples returned from Mars missions are categorised as Planetary Protection Category V, restricted as are missions returning samples from Europe or Enceladus. These returned samples are regarded as having the potential of containing life or signatures of life. The consensus is that such samples must be handled under BSL-4 containment until deemed to be free of any biohazard. The combination of current BSL4 practices with the cleanliness requirements of a curation facility will lead to the development of a new concept.

Contamination monitoring: When entering the curation laboratory, samples are considered pristine (i.e. they have not suffered terrestrial contamination), and thus, they should stay pristine during subsequent curation activities. In order to preserve the research value of these precious samples (i.e. terrestrial contamination would alter the scientific significance of these extraterrestrial materials), contamination, but also physical and chemical alteration must be minimized, understood and properly documented. All the curation operations should be carried out in positive pressure nitrogen, steel cabinets. If possible, it is also more than recommended to use different cabinets for different types of samples/missions and to also only work on one "parent sample" at any one time in a cabinet.

Manipulation of the samples: For the recovery of the samples from the sample container and transfer to the storage container, different methods have been used in the past, depending on the size of the sample, including picking-up directly the samples from the container, scooping the samples using a PTFE spatula or using the compulsory free-fall method. The recovery and manipulation of extremely small samples (a few micrometers in size) is very challenging. It could either be done manually or robotically, using micromanipulators (e.g. electrostatic manipulators). On top of the manipulations themselves, proper approaches of characterisation and storage of such small samples will have to be developed.

Characterization and Database: At first an initial processing and characterization of the samples should be conducted, including naming (a sample ID is given to each sample),

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photographing (such as basic 2D digital photographs and 3D laser scans), weighing and description of the samples (size, colour, etc.). These data should be directly entered in the specifically designed electronic database using the secured network available in the curation facility. A large number of additional information will also be stored in the database, including the history of the sample (transfer dates, name of the operator(s), type of manipulation, comments, etc.). The database, containing the entire processing history of each sample, will need to be adequately designed to be able to incorporate all the possible subsamples and also to be easily updated with new fields and functionalities.
Preliminary examination using different methods such as X-ray microtomography, (field-emission) scanning electron microscopy (SEM; equipped with focused ion beam), X-ray fluorescence, microRaman spectroscopy, etc. should be envisaged (knowing that the extent of the preliminary examination will have to be discussed and defined in accordance with requirements from the scientific community and from the WP4). All analytical data generated during these initial analyses, including images, spectra, etc. will also have to be stored in the electronic database. Such a detailed documentation and database will then allow "virtual-loans" (i.e. remote examination of the samples by the researchers), but also online selection of appropriate samples for research, before submission of a loan request. This would not only allow reduced handling and limit unnecessary manipulations, but it would also ensure that loan requests are dealt with in the most appropriate manner.

Preparation and Allocation of samples: Part of the curation consists in the preparation of the appropriate samples and allocation to the requestors/investigators. Due to the limited amount of samples to be returned (and curated), and because samples should be preserved for the next generations of researchers, a minimum (but sufficient) amount of sample will have to be granted (after approval of the allocation of the sample(s)). For this reason the samples will have to be subdivided, and this can be done using for exemple precision-cleaned hand tools, ultrathinning techniques, or using a saw operated without blade lubrication. In the latter case the friction and induced increase of temperature will likely affect and, to some extent, alter the sample. Small particles can then be separated either by hand picking with tweezers, micromanipulators, or by dry sieving. Newly generated subsamples are named in an appropriate way, using an extended ID (i.e. using the "parent sample ID" and an additional number and/or letter at the end). The best way of naming samples and subsamples will have to be discussed.
Special sample preparations, such as mounted samples, polished or thin sections, FIB foils, etc. will have to be performed in the facility; new techniques of samples preparation will have to be envisaged following requirements from the researchers community and from the WP4.
The samples are then allocated to scientists and are technically "on loan" for specific approved studies. Following the completion of these studies the samples should be returned to the facility together with the generated data (i.e. to be stored in the database). Importantly, the "returned samples" will not go back with the "pristine samples", they will have to be stored in a proper way in a distinct laboratory.
Specific transport sample containers/holders will have to be designed according to the requirements of the different types of analyses to be conducted (inputs to and outputs from WP4 and WP6).

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5. Short list of the identified main requirements and important issues to be especially investigated

*Curation of extraterrestrial samples requires an extremely clean environment (with particularly low levels of chemical and particulate contaminants and no biological or organic contaminants) involving the use of positive pressure isolators. BSL-4 facilities use negative pressure isolators to protect operators. In addition, in BSL4 facilities, operators wear positive pressure suits to handle the samples. There may also be difficulties in developing sterilisation procedures intended to inactivate non-terrestrial life-forms.

*Such a facility should be designed to preserve and protect the samples for generations to come, with all the involved implications, especially in term of maintenance and incompressible costs in the long term.

*The construction of the facility should be achieved at least a year before delivery of the first samples, to allow a proper training of the personnel, to test (using analogues) and practice manipulation and all other curation steps, and to write and refine the many necessary procedures.

*A large (TBD) number of the requirements should be based on needs from the scientific community.

*A need of a significant technological advance over the methods currently used, especially for the handling and preparation of the samples, is evident.

*Sampling and especially storing gas and all other adsorbed volatiles, preserving ice and temperature-sensitive mineral phases, and dealing with samples that may contain traces of extraterrestrial organic material or prove to be biohazards will be a big challenge. New approaches of samples curation and storage will have to be developed.

*For potentially biohazardous samples, previous studies have suggested extensive use of remote manipulation, double walled isolators containing inert gases and automated biobanks would be required for any future Mars sample curation facility.

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Work Package 4: INSTRUMENTS AND METHODS

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 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

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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 come 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.

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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

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- 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. fracturation 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

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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 analogs and witness plates need to be defined in interaction with WP5 (Analogs).

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 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, subsamples 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)

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- 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.

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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 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*

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(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) including morphological but chemical and structural information as well. 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 mm for SEMs) but significant sample preparation and handlings are 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

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expect instrumentation within ESCF and relevant of this category to be restricted to biosafety issues and to some extent to contamination control.

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 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),
- 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

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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.

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 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

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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.

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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 equipments 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 constitutes a dedicated deliverable.

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.

Small samples from the cosmic dust collection are handled under a high magnification binocular microscope. They are picked up from the silicon oil collectors either by hand or using a micromanipulator (for particules smaller than 10 microns) and rinsed from residual silicon oil in a hexane droplet. Thereafter they are characterized (shape, surface, bulk

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chemical composition) by optical and electron microscopic examinations. This procedure has been adapted in other cosmic dust laboratories, where micrometeorites are extracted from polar snow or ice. Note that most of the cosmic dust collected in the stratosphere remain in collectors and are kept for future generations. For the Stardust samples, micromanipulation is the rule for the extraction of aerogel keystones with embedded comet dust.

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.

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 achieved 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.

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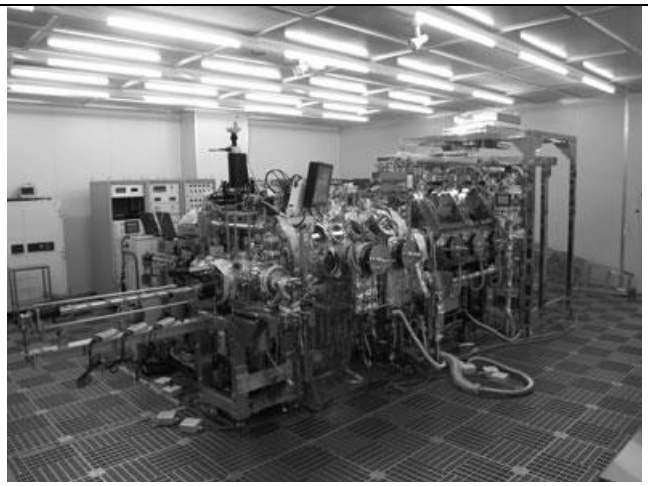

Fig. 1: Picture of the clean chambers in the JAXA Haybusa facility. They are composed of two main chambers located in a room with clean class level of 100-1000.

5.3. Other sample curation facilities

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. Because it is of potentially high interest for Euro-Cares, a description of the cryo-laboratory at University of Alberta is given here based on Herd et al. (2016).

Tagish Lake is an ungrouped type 2 carbonaceous chondrite with affinities with CI and CM chondrites. This meteorite was observed and found on the frozen surface of Taky Arm of Tagish Lake in January 2000. According to the pristine nature of Tagish Lake, a special cold room curation facility has been designed (The Subzero Curation Facility for Astromaterials at the University of Alberta). At the heart of the facility is an Ar gas glove box (MBraun, Inc.), housed within a controlled environment chamber capable of maintaining temperatures between -30 and -10 °C. The glove box—modeled after similar glove boxes at NASA Johnson Space Center and other curation facilities—consists of a single user station, 120 cm wide by 78 cm deep by 90 cm high, made of brushed 304 stainless steel with radius corners, with a polycarbonate window with chemical and scratch resistant coating. Integrated into the window is a binocular microscope (Leica, Inc.), fitted with a camera adapter. An adjustable stage sits beneath the microscope, within the glove box. Exchange of materials and samples into and out of the glove box is provided by either of two cylindrical, brushed 304 stainless-steel antechambers. The atmosphere within the glove box is maintained using an MB 20 G gas purifier (MBraun, Inc.); once charged with high-purity oxygen-free (99.998%) argon, the system continuously recirculates the argon through a purification system, which removes airborne contaminants and maintains O_2 and H_2O to <1 ppm. HEPA filters on gas inlets also

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reduce any particulate matter that may otherwise be circulated into the glove box. An activated carbon filter unit on the gas outlet for the main glove box removes any volatile organic compounds that might contaminate the materials used within the purifier; this feature also allows for organic solvents (e.g., chlorinated solvents such as DCM) to be used within the glove box, either for cleaning purposes or to carry out organic extractions on samples within a purified inert atmosphere at low temperature. Nearly all tubing used is 304 stainless steel; Viton^{TM} type B O- rings are used to minimize volatile compounds, similar to NASA JSC recommendations. The results of this curation are impressive. The contaminants within the Subzero Curation Facility for Astromaterials provide sufficient baseline information for the commissioning of the facility. Processing of Tagish Lake specimens now occurs on a routine basis. To date, no significant levels of organic contaminants have been observed in any meteorite samples, although the use of witness plates is planned but not yet implemented. In practicality, the facility accomplishes the purpose for which it was built, i.e., to enable the processing of Tagish Lake specimens under clean, cold conditions in an inert atmosphere.

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6- Future developments and next generation of instruments

An important point to be explored in work package 4 is to which 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

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interactions with space mission manufacturers.

7. Conclusions: baselines and key issues

In this document have been exposed the state-of-the-art in extra-terrestrial sample handling and characterization. We have identified current practices and presented key issues. We have tried to isolate the specificities of the EUROCARES requirements. We have prepared the next deliverables of WP4 in 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. One case might be an exception to that regulation. 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 curating 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 what extent of contamination assessment should be performed inside or outside the facility. While a minimum assessment should be done, possibly concerning mainly on-flight contamination, one would expect that contamination within the facility is minimum and therefore can be checked by the laboratories working on allocated samples.

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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Work Package 5: ANALOGUE SAMPLES

Analogue sites and analogue samples are used in space exploration for almost all critical steps between the start of a mission start to final sample analyses and data interpretation. They have proven important for various mission types, i.e. orbital, landing or sample return. For example analogue sites allow for testing landing and launch manoeuvers and rover mobility on extra-terrestrial bodies. On the other hand, analogue samples are widely used for testing calibration and functionality of remote instruments, as well as for interpreting data collected. If necessary they are used to carry out laboratory experiments in various domains, from planetology to astrobiology. In this sense, analogue samples are complementary to the classical calibration samples used for instrument development alone, for example, a colour target that is used to calibrate a camera or silicon used to calibrate a Raman spectrometer. In this document, both analogue and calibration samples will be considered.

The aim of the Euro-Cares project is to create a curation and analytical facility dedicated to extra-terrestrial samples brought to Earth from different bodies in the Solar System (Mars, the moons of Mars, asteroids, the Moon), either by unmanned and/or by manned missions. These samples will require particular storage conditions and handling procedures during curation and analysis. Analogue samples will be crucial in evaluating and defining the provisions necessary to accomplish safe and sustainable handling of extra-terrestrial materials. For example, they will allow for testing and improving the storage and handling container, sample preparation and analytical protocols. For practical reasons and sterility concerns, it might be necessary for the curation and analytical facility to have its own collection of analogue samples. The aim of this report is to list different types of samples that are required (analogues and standards), and to collate a preliminary list of analogue materials already available. This list will be completed over the course of this project in response to the requirements established by the other work packages, and might include recommendations for the fabrication of new artificial analogues.

While a human return mission could potentially bring back a few hundreds of kilograms of materials to the Earth (compare with Apollo missions on the Moon), it is likely that automated missions will bring back little material, on the order of a few grams and less (e.g., Stardust mission collecting cometary dusts). Thus, the storage facility should be flexible enough to deal with samples of different sizes and amounts. While large samples may be problematic in terms of storage and handling, very small samples are more challenging to study. The handling and preparation of very small samples can be difficult, espcially in sterile conditions. Moreover, the preparation required for some analyses must be associated with the least loss of material possible, and the analytical protocol must be very well defined in order to carry out the different measurements in a logical way. While it is obvious that the non-destructive analyses must be made first and destructive ones last, the protocol must also take into account the consequences of one type of analysis on another, as well as the potential intermediary preparation steps (coating and coating removal, for example). Analogue samples stored in the facility will thus permit: Analogue samples stored in the facility should permit:

- to test storage conditions and handling containers,
- to develop and improve sample preparation procedures (cutting, crushing, grinding, sieving…),
- to develop protocols for analysis,
- to support interpretation of instrumental limitations on analyses carried out on the “true” samples.

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Analogues for testing analytical procedures within the facility will also depend on the kind of instrumentation housed in the facility. While the basic characterisation of the samples will be undertaken in the receiving facility, it is expected that more detailed investigations will be made in individual laboratories, unless the samples host evidence of extant life, in which case they will not leave the facility unless thay have been throroughly sterilised, a procedure that could compromise certain types of analysis.

The different types of analogues can be categorised as shown in Table 1. In the framework of the EuroCares curation facility project, only analogue samples will be considered, not analogue sites or simulation chambers. More information about analogue sites can be found in Preston et al. (2012), Cousins et al. (2013), Cousins (2015), and Harris et al. (2015) for example.

Nature	Type	Relevance	Example
Natural analogues	*Site*	*Geology*	*Olivine rich sandy plains, Iceland (Mangold et al., 2011)*
		Geomorphology	*Mobility training in Utah desert, USA (Foing et al., 2011)*
		Processes	*Acidic alteration in Cyprus (Bost et al., 2013a)*
		Mineralogy	*Jarosite in Rio Tinto, Spain (Edwards et al., 2007)*
		Astrobiology	*Arsenic bacteria, Mono Lake, USA (Wolfe-Simon et al., 2010)*
		Test and calibration	*AMASE in Svalbard (Amundsen et al., 2010)*
	Geological sample	Geology	Impactite rocks
		Mineralogy	Anorthosite (Moon analogue)
		Cosmochemistry	Meteorites
		Astrobiology	Rocks containing fossils of anaerobic microorganisms (Westall et al., 2011)
		Test and calibration	Diamond
	Biological sample	Astrobiology	Extremophiles (Rothschilde and Mancinelli, 2002)
		Test and calibration	Various bacteria (Parro et al., 2008)
		Planetary protection	Various bacteria (http://planetaryprotection.nasa.gov/methods)
	Chemical sample	Cosmochemistry	Organic compounds in meteorites
Simulated	*Site*	*Test and*	*Lander touchdown and rover mobility (Richter*

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analogues		*calibration*	*et al., 2007)*
	Simulation chamber	*Cosmochemistry*	*Cometary analogue simulation chamber (Danger et al., 2013)*
		Test and calibration	*Mars 500 experiment in ESA*
	Biological sample	Astrobiology	Artificially fossilized microorganisms (Orange et al., 2009)
	Chemical sample	Cosmochemistry	Analogue of tholins, Titan aerosols (Carrasco et al., 2013)
		Astrobiology	Pigments for Raman spectroscopy (Vitek et al., 2009)
		Test and calibration	Pure molecules
		Planetary protection	Biomolecules
	Material samples	Test and calibration	Colour target for cameras
		Handling and transportation	Gas to test airtightness of a sample return container
		Planetary protection	Resins used for space probes

Table 1. Analogues and calibration samples sorted by nature, types and relevance. The analogue sites and simulation chambers (in italic) are not addressed in the framework of the EuroCares project.

1. Brief overview of existing sample receiving facilites

1.1 Planetary Material Sample Curation Facility of JAXA (PMSCF/JAXA):

The PMSCF/JAXA in Sagamihara, Kanagawa, Japan, was established to curate planetary material samples returned from space in conditions of minimum terrestrial contaminants (Yada et al., 2014). The first samples to be stored there were those from asteroid 25143 Itokawa, returned by the Hayabusa space craft. Before curation of these samples, the curation facility went through a series of comprehensive tests and rehearsals.

1.2 Extraterrestrial sample storage at the NASA-Johnson Space Center, Houston

A variety of extra-terrestrial samples is stored at JSC including lunar rocks, meteorites, cosmic dust collected in the upper atmosphere, cometary and interstellar dust from the Stardust mission, and solar wind particles from the Genesis mission

The mission Stardust to the Comet Wild 2 captured grains from the comet and interstellar dust. The contents of the Stardust Return Capsule, including the aerogel and the samples embedded in it, were maintained in an ISO Class 5 cleanroom environment throughout the initial sample processing. Particulate and non-volatile residue (NVR) witness plates were used to monitor the environment during the times aerogel was open to the laboratory air, and monitored daily for visible particulate contamination. The remaining portions of the SRC are

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curated in the Space-Exposed Hardware Laboratory for characterization of the effects of exposure to contamination and the space environment, including surveys of the micrometeorite impact record.

Photograph of the Stardust cleanroom setup (left) and the Lunar lab (right) at JSC

The Lunar laboratory at JSC provides permanent storage of the lunar sample collection in a physically secure and non-contaminating environment. The purpose of the facility is to maintain in pristine condition the lunar samples. The samples are stored and handled in stainless steel glove cabinets that are purged by high-purity nitrogen gas to minimize degradation of the samples. Pristine samples are always separated from human hands by three layers of gloves.

2. Samples required at for a curation facility

2.1 Geological samples

During *in situ* missions, a large part of the investigations made by rovers and landers are carried out on rocky samples *sensu lato* (*i.e.* including ices). Whether it is to study the geology, to search for traces of life or to search for organic compounds, the initial sample is either a consolidated rock or a grab sample of loose grains as e.g., regolith and soil on Moon or Mars. Several collections of geological analogue samples exist, such as the International Space Analogue Rockstore, ISAR, www.isar.cnrs-orleans.fr, (Bost et al., 2013b) or the different geologic, mineralogic and meteorite collections in natural history museums.

The table 2 lists some of the most common rocks found on the different bodies expected to be concerned by a sample return mission in the future. It will be necessary to have fully characterized analogue samples of these rocks as references in the facility. It is important to note that some of these samples are not available on Earth and must be synthetized. This list will be updated regularly following the new discoveries done on the different bodies. For example, in recent years, in particular following the results from the Mars Exploration Rovers and the Mars Science Laboratory, the concept of Mars analogues has changed and expanded. The MERS identified volcanic rocks and secondary precipitations, such as jarrosite and hematite (Klinglehöfer et al., 2004) MSL has identified fluvial, deltaic and lacustrine deposits of volcanic composition, with secondary salt deposits (Mg and Ca sulphates) in Gale Crater, and some relatively differentiated silica, alkali-rich igneous float rocks (Grotzinger et al., 2014; Sautter et al., 2014). As a result of the MSL findings, a wider range of sedimentary and igneous rocks are required as analogues than have been considered in previous analogue studies.

Rock type	Name	Body	Analogue type
Volcanic	Picro-basalts	Mars (McSween et al, 2009)	Natural

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rocks	Basalts	Mars (McSween et al, 2009)	Natural
		The Moon	Natural
		Asteroids	Natural
	Basalt andesites	Mars (McSween et al, 2009)	Natural
	Andesites	Mars (McSween et al, 2009)	Natural
	Basanites	Mars (McSween et al, 2009)	Natural
	Tephrites	Mars (McSween et al, 2009)	Natural
	Phono-tephrites	Mars (McSween et al, 2009)	Natural
	Trachy-basalts	Mars (McSween et al, 2009)	Natural
	Basaltic glass	Mars (Fabre et al., 2011)	Natural and synthetic
	Anorthosites	The Moon	Natural
Impact rocks	Basalt impactite	The Moon	Natural
	Impact melt rocks	Asteroids	Natural
Sedimentary rocks	Clays	Mars (Meunier et al., 2012)	Natural and synthetic
	Oxides	Mars (Calvin et al., 2008) Natural	
	Volcanic sediments	Mars (Vaniman et al., 2014) Natural/synthetic	
	Sulphates	Mars (McLennan et al., 2014) Natural	
	Carbonates	Mars (Boynton et al., 2007)	Natural
Soils	Moon regolith	The Moon	Synthetic (Willman et al., 1995; Carpenter et al., 2006; Hill et al., 2007; Schrader et al., 2010)
	Asteroids regolith	Asteroids	Synthetic
	Mars regolith	Mars	Synthetic (Allen et al, 1997; Vijendran et al., 2007)
Ices	Cometary regolith	Comet	Synthetic
	Clathrates	Mars	Natural/Synthetic

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	Mars permafrost	Mars (Smith et al., 2009)	Synthetic (Chevrier et al., 2007)
	Icy moons regolith	Titan	Synthetic
Meteorites	Chondrites	Asteroids	Natural
	Achondrites	Mars	Natural
		The Moon	Natural
		Asteroids	Natural
	Iron meteorites	Asteroids	Natural
	stony-iron meteorites	Asteroids	Natural

Table 2. Most common rocks on the different bodies expected to be concerned by a sample return mission and availability of their analogue (to be completed).

Complementary to this list, analogue samples of expected targets are needed for astrobiological reasons in particular (Table 3).

Type	Name	Body	Analogue type
Sedimentary rocks containing fossil traces of anaerobic microbes	Archaean cherts (Westall et al., 2011, 2015)	Mars	Natural
	Hydrothermal deposits (Callac et al., 2013		
	Salt deposits (Barbieri and Stivaletti, 2011)		
	Carbonate mudmounds (Marlowe et al., 2014)		

Table 3. Analogue samples of astrobiological interest (to be completed).

Finally, pure minerals can be required to calibrate instruments at the facility. The list of these samples will be defined in accordance with the list of available techniques however, it is still possible to establish a list a pertinent minerals (Table 4).

Class	Minerals	Found in/on	Useful for spectroscopy
Carbon	Graphite	Meteorites (Quirico et al.; 2009)	Raman spectroscopy
	Diamond	Meteorites	Raman spectroscopy
Silicates	Quartz	Mars (Blake et al., 2013; Bish et al., 2013)	Raman spectroscopy
	Olivine	Mars (Blake et al., 2013; Bish et al., 2013)	Raman spectroscopy
		Meteorites (Blake et al., 2013)	
		The Moon	

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		Asteroids	
	Pyroxenes	Mars (Blake et al., 2013; Bish et al., 2013) Meteorites (Blake et al., 2013) The Moon Asteroids	Raman spectroscopy
	Amphiboles	Mars (Blake et al., 2013; Bish et al., 2013) Meteorites (Blake et al., 2013) The Moon Asteroids	Raman spectroscopy
Iron oxides	Hematite	Mars (Bish et al., 2013)	Raman spectroscopy
	Goethite	Mars	Raman spectroscopy
	Magnetite	Mars (Bish et al., 2013)	Raman spectroscopy
Sulphates	Jarosite	Mars (Madden et al., 2004)	Raman spectroscopy
	Gypsum	Mars (Fishbaugh et al., 2007)	Raman spectroscopy
Iron sulphide	Pyrite	Mars	Raman spectroscopy
Carbonates	Calcite	Mars (Boynton et al., 2007)	Raman spectroscopy
	Dolomite	Mars	Raman spectroscopy
	Siderite	Mars	Raman spectroscopy
	Ankerite	Mars	Raman spectroscopy

Table 4. Common minerals useful for calibration and/or pertinent as analogue samples (to be completed).

2.2 Chemical samples

Some chemical samples will be required as reference materials and to test and calibrate the instruments. A non-exhaustive list is displayed in Table 5. However, since these sample types are generally less stable than geological samples, a large part of chemical analogue samples would be chosen for each sample return mission in preparation (see part 2.2).

Class	Molecule	Found in/on	Useful for spectroscopy
Amino acids	Glycine	Murchison meteorite (Cronin et al., 1985)	GC-MS IR spectroscopy
	Valine	Murchison meteorite (Cronin et al., 1985)	GC-MS IR spectroscopy
Sugar related	Glycolalde	Interstellar medium (Jorgensen	GC-MS

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compound	hyde	et al., 2012)	IR spectroscopy
Pigments	Beta-carotene	Living organisms	Raman spectroscopy (Vitek et al., 2009)
	Chlorophyll	Living organisms	Raman spectroscopy
Organic/ice mixtures	e.g.	Cometary (de Marcellus et al., 2015)	

Table 5. List of chemical analogue and reference compounds (to be completed).

2.3 Technical properties samples

In order to test the different instruments available in the facility as well as sample preparation systems, some test samples will be needed, such as materials with different technical properties (porosity, density, size, roughness…). The list of these samples has to be defined in accordance with the techniques available at the facility (to be defined). Table 6 shows some classical calibration samples.

Material	Used for
Silicon	Raman spectroscopy
Colour target	Camera
Density references	Preparation systems
Porosity references	Preparation systems
Weight references	Handling systems
Size references	Handling systems Preparation systems
Shape references	Handling systems Preparation systems

Table 6. List of calibration samples (to be completed).

2.4 Biological samples

Biological samples will be needed for astrobiological and planetary protection considerations. From an astrobiological point of view, certain types of biosignatures, such as extremophiles or other fossilised signatures of anaerobic microorganisms, would be pertinent for study in preparation of a sample return mission from Mars or from icy satellites of Jupiter and/or Saturn. Biological test samples, for example for sterilisation, stroage, handling and preparation procedures, would be mainly used for planetary protection considerations to determine whether extant life exists in the samples (from Mars). Indeed, it will benecessary to

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ensure no contamination by a potential extraterrestrial microorganism as well as to avoid any false detection of extraterrestrial life. The table 7 lists the type of organisms susceptible to be interesting to have at one disposal at the facility.

	Microbial genus or species	Phylum	Comment
Extremely resistant models used to test sterilization procedures	*Bacillus* sp.	B	Typical spore-forming lab models (Horneck et al., 2012)
	Clostridium sp.	B	Typical spore-forming lab models (Horneck et al., 2012)
	Desulfotomaculum sp.	B	Spore-forming, autoclaving resistant (Aüello et al., 2013; O'Sulivan et al., 2014)
	Xanthoria elegans	E or E+B*	Lichen, desiccation and ionization resistant (Onofri et al., 2012)
	Rhizocarpon geographicus	E or E+B*	Lichen, desiccation and ionization resistant (Onofri et al., 2012)
	Deinococcus radiodurans	B	Bacterial radioresistant model
	Thermococcus gammatolerans	A	Archaeal hyperthermophilic radioresistant model (Tapias et al., 2009)
Typical human body contaminants	*Micrococcus* sp.	B	Skin colonizer
	Proteus sp.	B	Skin colonizer
	Pseudomonas sp.	B	Skin and mouth colonizer
	Streptococcus sp.	B	Skin and mouth colonizer
	Staphylococcus sp.	B	Skin and respiratory tract colonizer
	Escherichia coli	B	Gastrointestinal colonizer
	Malassezia sp.	E	Skin colonizer
	Geotrichum sp.	E	Mouth colonizer
	Candida sp.	E	Mouth colonizer
Anaerobes	*Halophiles*		
	Psychrophiles		
	Thermophiles		
	Acidophiles		
	??????		

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Table 7. List of typical microbial models in planetary protection used either to test sterilization procedures or to detect human contaminants (to be completed).

As a conclusion of the part 1, it is important to note that the amount/number of required samples will be variable depending on the process being tested. For instance, Dyar et al. (2015) used more than 3,500 pressed pellets of rock, mineral, and chemical standards for calibrating Laser Induced Breakdown Spectrometer.

3. Implications on the facility requirements

This section concerns both analogue samples as well as calibration samples as they will probably be stored in the same place. This will imply requirements in the curation facility.

3.1 Geological samples

Most natural geological samples have been exposed to atmospheric conditions for several thousand years or more and, thus, there is no particular requirement regarding their storage. However, they should be sterilised before using them for testing and calibration of the instruments.

However, samples that can oxidise in the atmosphere (Fe-rich rocks, for insatnce) or meteorites should be stored under controlled atmospheres. Similarly, some artificial samples will need particular storage conditions. Finally, ice analogue samples must obviously be stored in cold conditions.

A room dedicated to sample preparation is also needed to test different protocols: crushing, sieving, cutting, grinding, thin section preparation… Some standard instruments such as a polarized optical microscope will be necessary for rapid observations.

3.2 Chemical samples

Chemical samples require particular storage conditions. A well ventilated and relatively clean room is required.

3.3 Biological samples

A dedicated room is needed to store cellular cultures in cryopreservation agent (PEG) at -80°C.

3.4 Technical properties samples

The technical properties samples would be stored in different locations depending on the which instrument they would serve

3.5 General requirement

The different samples available must be referenced in a database with their associated characteristics, relevance, size, mass…

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Work Package 6: PORTABLE RECEIVING TECHNOLOGIES

1. Introduction

1.1 Aims and Objectives

Objectives: to propose methods for recovery and transport of Mars Moon or asteroid samples from the landing site to the curatorial facility.

This study consists of a detailed literature review and knowledge capture exercise for Portable Receiving Technologies. The status, mission architecture and science objectives of potential sample return missions to asteroids, the Moon and Mars. A preliminary report will be generated for each theme highlighting the requirements and important information identified during the knowledge capture.

Specific Objectives of the work will be:

- To determine what information and procedures are necessary for preparation for recovery of the sample
- To assess what tasks and facilities are necessary for recovery and initial inspection of the sample
- To determine how the procedures for recovery to be used will differ in the case of i) Mars samples (which contain the risk of biohazard) and ii) Lunar/asteroid samples including ice-bearing ones.
- To provide a concept for the transport of the sample to the curation facility
- To outline needs for innovation, particularly in terms of portable receiving facilities and transport
- Assess legal issues & public concerns associated with the transport of potentially hazardous sample containers.

1.2 WP6 Portable Receiving technologies description

The objective of this work package is to propose methods for the recovery and transport of Mars or Lunar/asteroid samples from the landing site to the permanent curatorial facility. The Earth re-entry capsule from a sample return mission will be targeted at a specific landing ellipse on the Earth, possibly a considerable distance from the curatorial facility. Before the capsule arrives, considerable preparations for the recovery need to be made. Once the capsule has landed, an assessment of the state of the spacecraft will lead to a recommended recovery procedure. The sample will then be transported to a permanent curatorial facility using a safe and secure method. This is covered in Figure 6-1.

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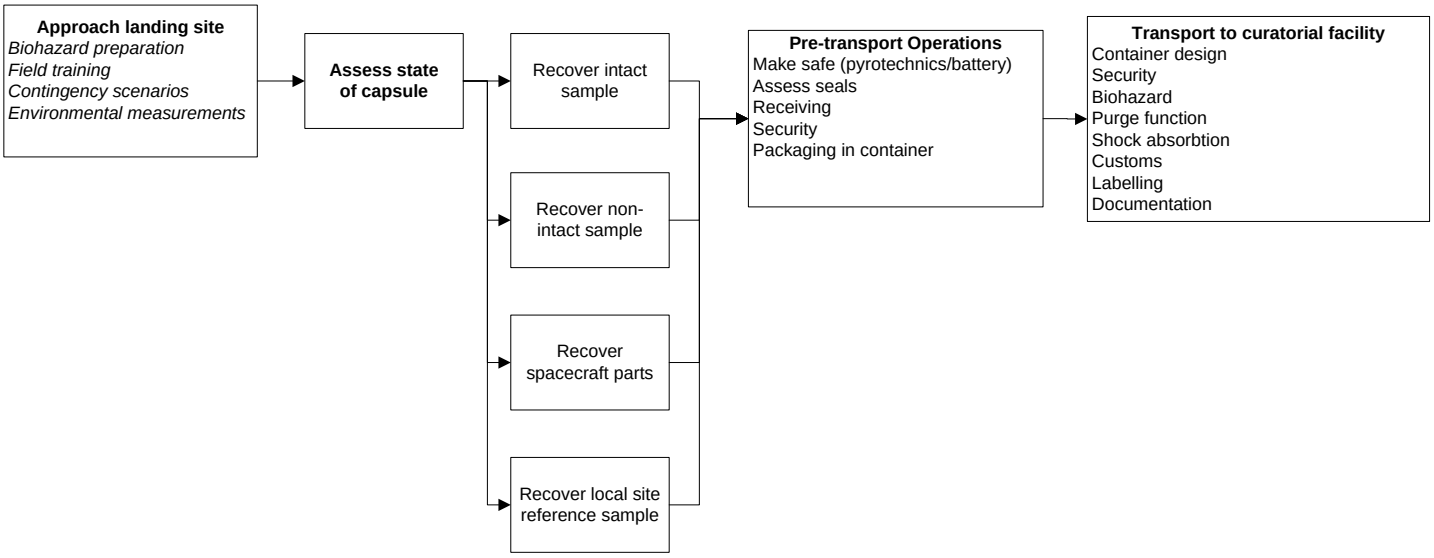

Figure 1-1 Flow diagram to show recovery of sample return capsule to curatorial facility

2. Review of previous spacecraft sample return recoveries

2.1 Genesis

The Genesis Return Capsule, bearing the science canister with collected solar wind samples, returned to Earth in 2004. Following a flawless, on-target re-entry the parachutes failed to deploy due to a set of incorrectly oriented deceleration sensors. The spacecraft impacted the landing site – in the Utah Test and Training Range (UTTR) – at a speed above 86 m/s and was badly damaged (Fig. xx). Most of the fragile collectors were fractured and all were contaminated on the surface by debris from the spacecraft and the landing site. A dedicated team of spacecraft engineers and curators immediately went to work to recover the broken spacecraft and move it to a temporary cleanroom at UTTR, where they painstakingly packaged and cataloged thousands of spacecraft parts and collector fragments. These were transported to the Genesis Curation Laboratory at JSC for cleaning, documentation, storage, and allocation. It is believed that all of the collector materials were recovered.

Figure 2-1 : Genesis capsule recovery (Image credit : NASA)

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2.2 Stardust

The Stardust Sample Return Capsule (SRC) was released from the mother spacecraft, and parachuted to Earth at UTTR in the early morning hours on January 15, 2006 (Fig. 15). A significant problem during the recovery was that the SRC landed upside down, which severely limited the usefulness of the recovery beacon. Once on the ground, the Stardust SRC was recovered by a team of curators and spacecraft engineers within 2 h, and was moved to a class 10,000 (ISO class 7) modular cleanroom located in a facility close to UTTR for preliminary processing (Zolensky et al 2008). The science canister was removed and secured in a clean transport container in this facility. A significant recovery flaw was that the SRC was placed into a polyethylene bag for several hours, and outgassing from this bag contaminated the aerogel capture media with several organic molecules (Sandford et al., 2006, 2010). Following the preliminary processing, the SRC was placed into a dry nitrogen environment and flown to the Stardust Laboratory at JSC in a specially chartered plane. The Stardust Science Team used a class 100 (ISO class 5) cleanroom at JSC for preliminary examination and curation of the returned samples Logistics associated with receiving these samples required careful planning and coordination with JSC Receiving, Security, Safety, Quality Assurance, Photography, and Curation. The samples received a police escort from Houston's Ellington Airport to the curation facility at JSC (Zolensky et al 2008).

Fig. 2-2. Stardust sample return capsule at Utah Test and Training Range recovery site.

2.3 Hayabusa-1

Following a series of propulsion, communication, and control failures, the spacecraft successfully returned to Earth in June 2010. The return capsule was predicted to land in a 20 km by 200 km area in the Woomera Prohibited Area, South Australia (Figure 2-3).. Four ground teams surrounded this area and located the re-entry capsule by optical observation and a radio beacon. Then a team on board a helicopter was dispatched. They located the capsule and recorded its position with GPS. Following ensuring that batteries used with EDL were safe and disconnected, the capsule was placed into a container with a nitrogen atmosphere, for transportation, initially to a temporary facility in South Australia.JAXA built and equipped a main laboratory in Sagamihara, Japan to carry out the external cleaning and de-integration of the recovered spacecraft, sample extraction and preliminary examination, and sample curation for the Hayabusa mission. This is the first non-NASA or Soviet facility for curation of samples returned from space.

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The returned hardware was planned to include one sample of ~100 g, but due to the failure of the sampling system, only ~1500 grains of asteroid material were recovered. These are still immensely valuable scientifically, and were recovered from the sample container on an individual basis. Contingency facility operations were needed where micromanipulation was used to sort genuine asteroid particles from contamination particles.

Figure 2-3 : Hayabusa sample return capsule after landing in Australia (Left) and Hayabusa sample container prior to opening (right) Image credit : JAXA

2.4 Osiris-REX (NASA)

OSIRIS-REx (Origins, Spectral Interpretation, Resource Identification, Security, Regolith Explorer) is a NASA mission slated for launch in 2016 to encounter and sample Asteroid (101955) 1999 RQ36 and return ~60g back to Earth. The sampling is based on a "Touch-and-go" method that will retrieve sample directly off the surface in a single collector (Figure xx) and return it to Earth in a return capsule similar to that used by the Stardust mission.

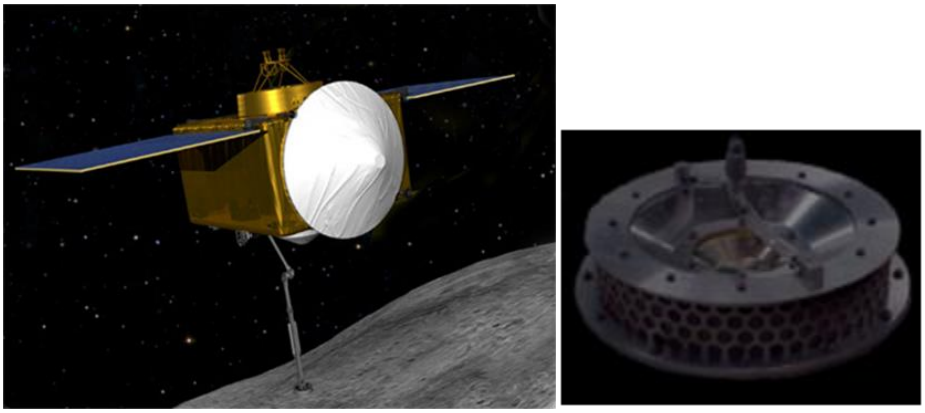

Figure 2-4 : OSIRIS REx spacecraft sampling an Asteroid and TAGSAM sample collector (Credit: NASA/GSFC/UA)

3. Current designs for Mars Sample Return missions

The current MSR mission scenario has an 'Earth Return Capsule' (ERC) which performs a hard landing at a sparsely occupied location on Earth. Inside the ERC is a biocontainer (BC). Inside the Biocontainer is a Sample Container (SC) and inside this are the Sample Vessels (SV). The exact amount of sample and number of sample vessels is subject to change.

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The outside of the hardware down as far as the biocontainer (BC) (see *Figure **3-1***) is considered to be Earth contaminated during landing and so high level contamination protection are in theory not needed. However if the ERC is breached or damaged in some way, contingency measures may need to be in place.

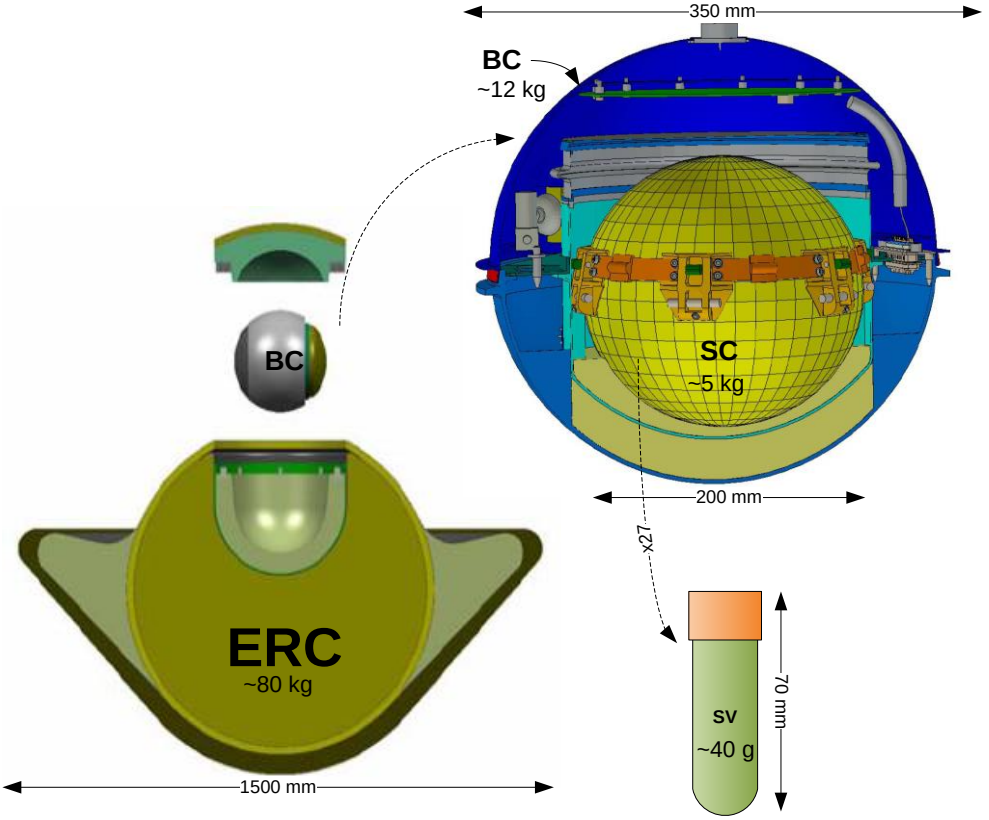

Figure 3-1 : BioSMoS (Bio-Sealing and Monitoring Technologies for Sample Containers) Concept

As a comparison it is useful to look at the Life Marker Chip project sample chamber (SPS). Meeting the PP requirements of category IV missions, the SPS was designed to accept a small sample (approx. 1cm^3) through a 5 mm aperture into a sealed and sterile Ti chamber. The flight representative model in the fig below is not ideal for a MSR sample; however, it serves to illustrate the engineering constraints of such a chamber (Figure 3-2).

Figure 3-2 : University of Leicester Life Marker Chip Sample Processing Chamber

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4. Preparation for Recovery

4.1 Introduction

A landing site is ultimately dictated by orbital mechanics, spacecraft design and the mission architecture. By comparison with missions like Genesis, Stardust, Hayabusa and Osiris-REX, mass and physical size is likely to be very different. Hayabusa, for example, was designed to return a single sample of approximately 100g, which in turn, dictated the service requirements and hence, volume of the landing component. NASA's Mars2020 mission is currently being designed as the first stage of a MSR mission where a subsequent retrieval lander / spacecraft will collect its cached samples. (Obj C of the Mars2020 SDT Report). For this reason, an estimate of sample size is based on these mission requirements. Section 6.2.3.1 of the SDT defines a total sample mass of 500 g divided over approximately 31 individual samples, which gives a sample mass of between 15 to 16 g each. It is also assumed that a sample may contain rock cores, regolith, ice, brine and gas.

In particular, MSR will represent a considerable investment with each sample having a substantial dollar value per gram of material. Risk management, from the point of view of sample integrity, will therefore influence the design of the sample container sub-system. Additionally, the number of discrete sample containers and the engineering requirements for each sample will ultimately dictate the mass of the Earth landed component. The sample return capsule of Genesis had a total mass of 225 kg and failed to land successfully, due to incorrect operation of accelerometers. This strongly suggests that that for a complex mission, such as MSR, the landed component is unlike any previous return mission and perhaps closer in design to a Soyuz type module.

4.2 Landing site

In terms of a landing site, the final selection will be driven by both the scientific requirements of the samples and the small risk of backward contamination of the immediate area. As discussed above, the return capsule of a MSR mission is likely to be larger than any previous return programme and will limit landing sites. Not considering the energy / orbital constraints of a Mars to Earth return spacecraft, there are two main considerations in terms of a landing site.

Security of the site (inc. contingency for a failed landing)

Accessibility of the site, in particular if specialist recovery vehicles are required

It is conceivable that NASA might use components of its new Orion spacecraft to test capability in the return of samples. This being the case, many of the landing site issues, security, safety and risk in particular will have been resolved.

4.3 Environmental conditions

Irrespective of the sample environment during the return journey, the ideal conditions to limit the risk, albeit very small, of any pathogen in a failed landing situation, is cold and dry. This will slow any chemical reaction rates with the local environment. From a sample integrity perspective, it will also be important to protect the samples from the local environment in the event that seals are compromised and cold dry desert type areas tend to be relatively sterile.

4.4 Assessment of the state of the capsule

The landing outcome will dictate two distinct management strategies.

- Successful landing

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- Failed landing (not managed like an unplanned event)

Except for an obvious failed landing, protocols must assume that at least one seal is compromised until proven otherwise. (These protocols need to be assessed)

Due to the increased size and mass of MSR, it is not possible to simply pick up the module as might have been possible with missions like Hayabusa (mass 18kg). The restrictive nature of working in a BSL4 suite must also be considered as a limiting factor, both in terms of time, cost and capability. Therefore, it is strongly suggested that any return module be fitted with a post landing EGSE connecting interface such that certain parameters (e.g. seal integrity) can be checked and monitored. This facility enables situation management; for example, if seals are all determined to be satisfactory, recovery might continue with reduced bio-safety protocols.

4.4.1 Integrity of Seal

As stated in the 2009 NASA Assessment of Planetary Protection Requirements for Mars Sample Return Missions; "…a critical issue …concerns the means by which those charged with implementing a Mars Sample return mission can demonstrate the integrity of the canister's seal."

Seals largely fall into two main types; static (examples being an O ring or metal gasket) or dynamic (an example being a lip seal). Static seals tend to be used where there is a requirement to provide a physical barrier between relatively non-moving interfaces such that the physical content of each side are kept separate. Interaction may still occur in some circumstances if the respective environmental conditions, a temperature gradient for example, are transmitted through the seal medium; which also has to be a design consideration. Dynamic seals differ in that they provide the same isolating function as a static seal, but are required to permit relative motion between the seal and the sealing surface interface. However, dynamic seals often utilise a lubricating fluid film between the seal elastomer and the sealing surface to reduce friction (typically <0.5µm). Lubricating films cannot be used in a MSR seal.

It seems likely, that the planetary protection requirements of any MSR container will require a combination of both static and dynamic sealing techniques, which in turn, drive the engineering requirements of that container. Of particular concern is the material selection, which is criterial both in terms of compatibility with the sample and the harsh environments of Mars, interplanetary (radiation effects on the seals) transfer and landing (mechanical shock). Hence, scientific integrity of the sample and the environmental conditions will drive the design of the seals, which in turn, will have significant implication on the mass, volume and complexity of the sample chamber sub-system.

One study [Younse et al, 2012] discusses four different sealing technologies (Teflon plug, crimp, solder & shape memory alloy) in terms of power, some environmental conditions, tolerance to dust, shock, integrity of the sample, hermeticity, packaging, risk and autonomy. A Teflon plug appears to be the most promising solution. Teflon, also known as polytetrafluoroethylene, PTFE, is the subject of a recent NSTP-2 investigation (University of Leicester) that will consider a different design approach to that given by [Younse et al, 2012]. The use of PTFE to form the seal body is considered for several of its mechanical properties, in particular a low coefficient of friction. Low van der Waals forces make the surface inert, due to the very strong carbon-fluorine bonds and therefore non-reactive to most other compounds. PTFE is also very hydrophobic, which prevents wetting by water and water based chemistry, an advantage if this technology was adopted in a "wet" chemistry application.

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However, a drawback of PTFE is the phenomena of creep (cold flow) where applied stress (force acting on the material) causes plastic deformation of the material. This must be a design consideration when the seal geometry is considered.

A critical design feature of PTFE tip seals is the surface finish of the sample chamber. An elastomer seal is able to accommodate a "rough" surface by deforming into the surface voids. The elastic properties of PTFE are low and require a high point contact stress to achieve a good seal (force often provided by a spring). In a dry dynamic application, this high contact stress is offset by the low coefficient of friction and benefits from an almost "optical finish" on the sealing surface. This has the added benefit that microbial cleaning is easier to attain.

Scientific integrity of the sample is crucial in that both the sample chamber walls and the seal material must be inert to the sample for the duration of the mission, which could potentially be 10 years. Two materials are often considered for the chamber; titanium (as is used in the LMC [Sims, Cullen and Holt, 2012] sample chamber) and gold, which are required, with the right surface finish, to exhibit either none or low absorption of organics, particularly important if an ice / water / brine based sample is acquired. Gold alloys are common in the electronics industry and there has been considerable investigation of their mechanical applications in high reliability swipe contacts. Pure gold is generally inert to most environments but exhibits relatively poor wear performance, tending to gall under high contact stress conditions. "Hard" gold is an alloy including other elements like 0.7% cobalt or nickel and with appropriate thickness (to overcome porosity) can provide a hard wearing sealing surface when used with a nickel transition layer. L168 aluminium alloy, with its increased strength and hardness, compared to many Al alloys, needs to be compared to stainless steel in terms of forming a sealing surface and considered as the base metal for a gold plated sample chamber.

As part of this study, it will be necessary to conduct an FMECA of the sampling and process of sealing the sample based on a risk analysis of sealing technology and a typical MSR architecture. This will enable weak points in the sampling chain to be identified and recommendations to inform the detailed design, which in turn will influence recovery. For example, many sample chamber designs seem to be based on a process whereby the chamber is not fully sealed before the sample is deposited; particularly the carousel type designs where a push type seal is applied after [Backes et al, 2012, Zacny et al, 2011]. As a category V mission with the intention of identifying very low level potential organic biomarkers, it is crucial that the sample chamber is pristine at the point of sample delivery; this was a space agency requirement on ExoMars.

[Guest and Bridges, 2011], identifies explosive welding as a potential high integrity sealing technology to achieve the leak rates that will be required for MSR. Other technologies including brazed and soldered metallic rings might be used in conjunction with the PTFE seal to achieve the high level of hermeticity required.

4.4.2 Other damage

It is a requirement that chamber integrity is monitored during return journey and landing. This could be achieved with a leak detection configuration or pressure sensors in the chamber [6]; either technique would require the chamber to be back filled with an inert gas. If the landed module included an EGSE connector point, it would be possible to verify the seal integrity and potential damage after landing.

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4.5 Special measures for biohazards

Any return of material from a mission to a planet thought capable of containing life would be carried out in such a way as to avoid uncontrolled release of a potential biohazard on impact with the earth. This would be a major requirement to protect the scientific purpose of the mission and also to prevent the potential release of extraterrestrial biohazards. Any uncontrolled release would be a low probability but high consequence occurrence. Therefore planning for non nominal returns which may lead to release of Martian material would be warranted and a precautionary approach would be taken

4.6 Planning for Non nominal return

Scenario planning will need to be carried out in order to identify the most effective way of dealing with a non-nominal return. The plans will need to take account of the following drivers

- Protection of the environment from release of Martian material
- Public perception of an environmental release
- Protection of the science
- Environmental protection from the impact of any remediation exercise
- Safety of the remediation workers
- Financial

These drivers will not work in concert. For example, a potential option to inactivate Martian material may be to generate a high temperature fire in the surrounding area using air dropped incendiaries. This would help to protection the environment from Martian material, allay public concerns and protect workers but would destroy the science and potentially damage the environment

Remediation after incidents involving biohazardous agents can vary from minor use of disinfectants to the removal of material for incineration. The recently published UK Recovery Handbook for Biological Incidents provides a decision making framework for dealing with environment contamination with biohazardous material can be made taking in account various factors (Pottage et al (2014)

An agreed method for decontamination of a Martian life form would need to be agreed before any return. This will inform the response.

4.6.1 Initial Approach to Returned Container

Unless sensors are embedded in the return canister to detect leaks or loss of pressure it will be difficult to assure that the returned container will be undamaged and any biohazard contained. Therefore some other method of identification of non-nominal return will need to be used This may be done using remote observation but if this cannot be done with confidence a robotic system could be used or a person wearing protective equipment could approach the landing site.

4.6.2 Non nominal return

If a non-nominal return is reported then an assessment of the required course of action must be taken based on the damage reported. The area would need to be secured and entry by unauthorised personnel prevented. The container should be moved into a contained space as soon as possible where it can be more closely observed and cleaned/decontaminated. A HEPA filtered space under negative pressure would be suitable. It would need some means to clean/decontaminate and some system to stor or inactivate any waste

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4.6.3 Conclusions

Planning of any sample return from a planetary body with potential for life will need to be carried out using worst case scenarios to ensure that preparation can be made for all eventualities to protect both the science and the planet.

5. Recovery and initial inspection

5.1 Introduction

Experience from the recovery of sample return missions to date show the importance of examining the entire sample handling and containment chain, including « landing site characteristics, ground recovery and transport to ground facilities, not just the quarantine or containment laboratory » (NRC, 2009)

In this section, the recovery and initial inspection of the sample will be covered, with recovery of spacecraft parts, portable laboratories, the challenges of handling and the public perception of risk examined subsequently.

5.2 Recovery of an intact sample

Previous missions have used different models of recovery:

Mission	Recovery
Genesis	Transport to temporary cleanroom at UTTR then on to Curation Lab at JSC
Stardust	Transport to class 10000 cleanroom at UTTR then on to JSC in plane
Hayabusa	Woomera and flown to Curation facility at Sagamihara

Both Stardust and Hayabusa-1 were recovered intact – although it is to be noted that even in these recoveries, there were incidents which risked compromising the science return.

After the Hayabusa-1 landing, the capsule was packed into a double layer of plastic bags filled with pure nitrogen gas and then inside an initial/temporary transportation box. The recovery capsule was then transported to the WPA Instrument Building where the recovery team and Quick Look Facility (QLF) were installed. One day was spent safing the explosive devices and the battery in the capsule. The next day was spent on the removal of contaminants adhering to the capsule and the packing the capsule into another clean transportation box for internal transport. The surface cleaning of the capsule and packing operation were both executed in the temporary cleanroom at the QLF installed in the building (Abe et al, 2011). The transportation box has a purge function of pure nitrogen gas, and can ease the shock under transportation. Then the capsule was put inside a cargo container which had air suspension to keep the capsule below 1.5 G shock during transportation [Matsuda, 2015] and then flown to the curation facility in a chartered plane.

Once Stardust touched down, a recovery Team was sent to find and collect the capsule. Depending on ground and weather conditions, the Recovery Team were planning to travel to the SRC landing point by helicopter or by 'MATTRACK' (a pickup with wheels replaced by treads). The recovered SRC was then transported to a cleanroom at the Avery Complex where the sample return canister was separated from the heatshield and backshell (Sandford et al, 2006).

To date, no sample return teams have set up a portable facility at the landing site. For a Mars Sample Return mission there may be a public perception that this is necessary in order to contain any potential contaminants if the spacecraft is damaged on reentry/landing.

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5.3 Recovery of a non-intact sample

5.3.1 Recovery

The Genesis recovery provides an example of a non-intact recovery as the Genesis capsule broke open on impact, and part of the inner sample capsule was also breached. This experience underscored the value of teamwork and contingency planning, and provides a vital set of "lessons learned" for future sample return missions (Ryschkewitsch, M., 2006).

The Genesis crash underscored the importance of thinking through multiple contingency scenarios and practicing field recovery for these potential circumstances. Having contingency supplies on-hand for all recovery operations was judged to be critical (Zolensky, 2008).

5.3.2 Decontamination of the area

The techniques used to decontaminate the landing area will have to agreed in advance by public health experts. Assumptions will have to be agreed on the potential resistance of biohazards to potential remediation options such as heat. Once a technique has been recommended then planning for its use can be started. The area could either be decontaminated or contaminated material could be removed for off site processing.

5.4 Existing portable laboratories

Truck and container labs are in use for outbreaks, environmental accidents and counter-terrorism. These containers can be loaded onto C-130 cargo planes or similar air transport and airlifted to the main laboratory. Examples are shown in Figure 5-1. A team who set up an on-site portable laboratory for a Marburg virus outbreak in Angola reported that the greatest challenge was the lack of consistent electrical power, this necessitated portable generators and battery backup systems for thermocyclers and the storage of samples at freezing temperatures was not possible. (Grolla and Jones, 2011).

Figure 5-1 : Germfree Mobile Container laboratories can be loaded on an aircraft, pulled as a trailer and transported by sea or rail. Image: © Germfree.

Another interesting concept used to investigate outbreaks of infectious pathogens up to the highest risk group 4 is a deployable mobile laboratory. This 'European Mobile Laboratory' was developed by the Bundeswehr Institute of Microbiology (InstMikroBioBW) in Munich, Germany, and can be stored and deployed on conventional transport (see Figure 5-2) (Stoecker and Woelfel, 2014).

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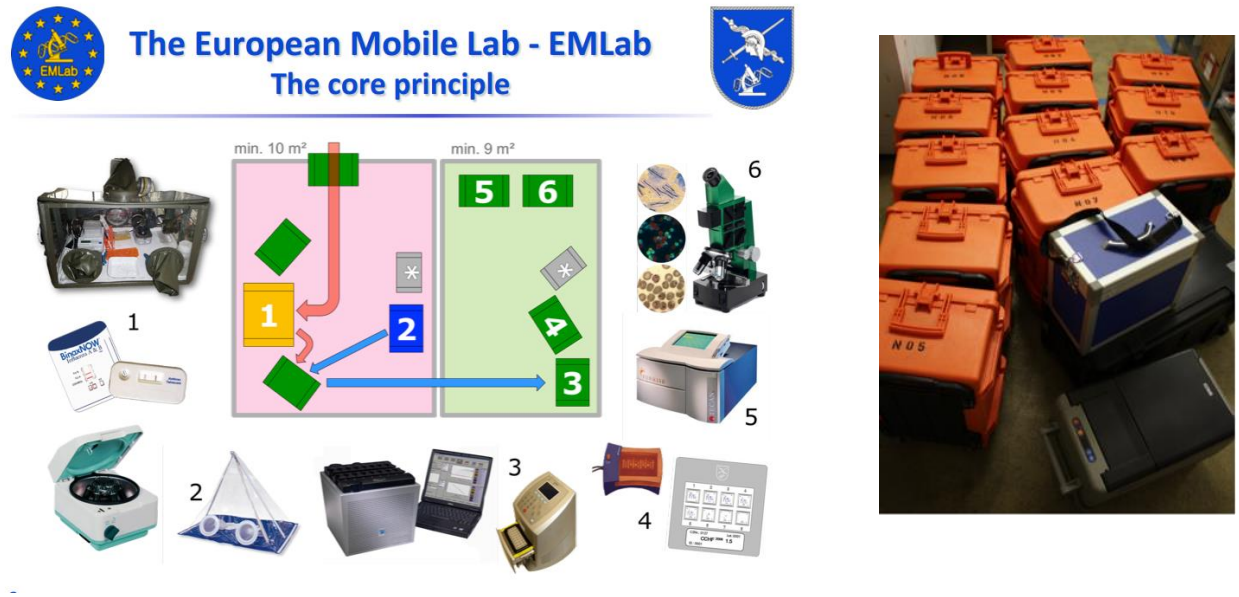

Figure 5-2 : European Mobile Laboratory is packaged in 15 easily transportable containers (Stoecker K. and Woelfel R., 2014).

5.5 Handling

Bridges and Guest, 2011 discuss potential sources of damage to a Mars Return sample once it has landed. These include :

5.5.1 Vibration and Shocks

The handling of the sample should not introduce vibrations or shocks and these could destroy any structure within the sample.

5.5.2 Electromagnetic Contamination

Any strong electromagnetic fields may compromise the electromagnetic properties of the sample and any static charge induced may allow a dusty sample to cling to surfaces which could make extraction challenging.

5.5.3 Orientation

In order to maintain the structure of the sample (which is useful for sedimentatry rock analysis), it may be advisable to retain the landed orientation during handling.

5.6 Perceived Risk and public perception

The ESF ESSC report (ESF-ESSC Study Group, 2012)discusses perceived risk and public perception of risk. It discusses the hazard vs the risk and the event chain necessary for substantial environmental consequences. In the same report it is suggested that «potential release scenarios are defined and investigated» in order to develop ways to respond.

6. Transport to / from curation facility

6.1 Introduction

The most important concept of the sample container is to deliver the small samples safely, with prevent them from terrestrial contaminants during its transportation. In order to reach

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this goal special precautions must be taken into account in the design and procurement of all containers (temporary or permanent) with which the samples will be in contact.

The recovery of the samples will be performed following several steps:

- Operations and packaging of the capsule on the landing site
- Operations in a temporary clean room (cleaning of external surfaces; check of integrity, ecc.)
- Operations at the curation: recovery of the sampling chamber, inspection and storage of the samples.

The delivery of the samples to scientific laboratories needs the definition of the packaging necessary to preserve its integrity during the shipment. In this case containers designed under the responsibility of the curation facility can be used as the standard delivery packaging.

However specific requirements in the samples preparation for analyses, requested, could require the adoption of different packaging. In this last case the responsibility of the package realization and/or procurement is under responsibility of the scientific laboratory requiring the samples.

6.2 Packaging

Packaging is a fundamental process, since it is aimed at minimizing possible sources of permanent damage, e.g. physical shocks, temperature change and humidity.

6.2.1 From landing site to Curation

At the landing point the capsule will be placed into a temporary plastic bag and a stored in a transportation box. The performances both for the temporary plastic bag and the transportation box have to satisfy mainly the following requirements:

- Guarantee a good insulation by the atmosphere
- Guarantee a good insulation from particulate and molecular matter
- Avoid organic contamination
- Preserve integrity of the capsule

A first check of the capsule and/or of the sample container integrity and a cleaning of external surfaces will be at the portable laboratory. After these operations a new transportation box will be used with the same protection capabilities of the previous one. In this case the package must have an improved performances of monitoring and a better sealing capability in order to preserve the samples during the transfer to the curation facility.

Basing on experience of previous sample-return missions, the recovery and transportation of the return capsule has not required extraordinary handling measures or hardware, due to the small size and mass of the return capsule, but has been obtaned simply by a specialized handling fixture to cradle the capsule during transport.

In particular, the Hayabusa samples were packaged in a container, sealed with double Viton O-Rings. This was not sufficient to avoid leakage of terrestrial air, which increases with time. Therefore, in order to minimize this effect, the sample container was placed into a ultra pure nitrogen atmosphere (Abe et al. 2011). The scheme of the Hayabusa sample container is given in Figure 6.1 (top).

The Hayabusa-2 sample container will be based on the same design of the Hayabusa one. The improvenet which will be applied will concern :

- Aluminum metallic vacuum sealings, with mechanical latching mechanisms

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- Noble gas ventilator at the bottom of the canister
- A larger Canister Volume (48x48x57.5 mm)
- Total mass lower than 500 g.

The scheme of the Hayabusa sample container is given in Figure 6.1 (bottom).

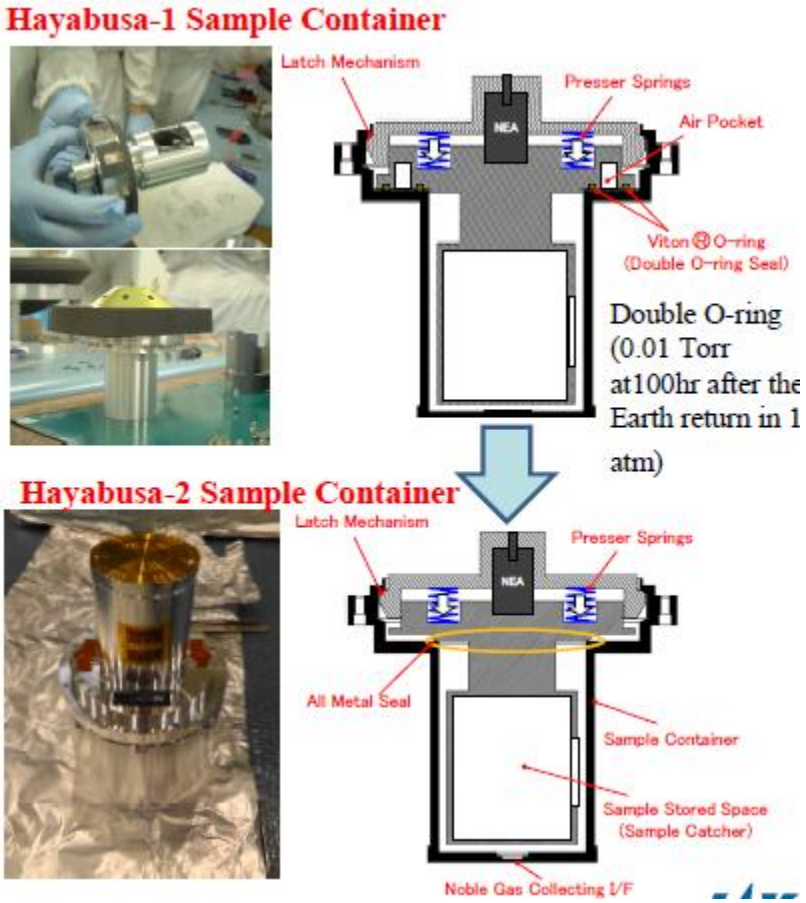

Figure 6.1. Hayabusa (top) and Hayabusa-2 (bottom) sample container.

6.2.2 From curation to Scientific laboratories

The transport from Curation to laboratories is generally allowed only after approval by the Curator.

Typical size of the sample is less than 100 μm, so handling of the sample is performed with a micro-manipulation system in the clean chamber of the curation facility (see the Stardust and the Hayabusa examples). The sample container for shipment will be able to preserve the sample from the contamination and guarantee the localization of the sample. An example of this kind of container can be the package used for the Itokawa samples collected by Hayabusa-1 mission.

The container consists of a pair of outside flanges and a pair of quartz glass plates. The flanges are made by stainless steel, the same material of the clean chamber. The base flange has been machined to be able to hold a pair of glass plates with clips and screws. A copper gasket coated with gold is set between a cover flange and the base flange. The pair of flange is then enclosed with six screw bolts to seal the pair of glass plate inside. Inside the pair of flanges, the base plate made of quartz glass is set to contain a sample particle. It has three to

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five dimples whose aperture is about 1mm and depth is less than 0.5mm. The samples are placed in those dimples one by one with the micro manipulator electrostatically controlled in the clean chamber. The metal plate is set under the base glass plate in order to increase the ability of the control the handling of the sample with the micro manipulation system. As the sample is set inside the dimple, a cover plate made of quartz glass is put upon the base plate though which the sample can be observed. The base plate is held with a metal clip and a screw and the cover plate is held with two pairs of a clip and a screw.

6.3 Customs and Regulatory issues

This will depends on the two countries involved ; the country where the landing site is located and the curation facility country. If the US has major involvement in the mission, then it is expected that ITAR restrictions will limit the landing site location to the US. If not, then the strongest candidate for the landing site would be Woomera, Australia. This area needs further investigation. In the case of Hayabusa mission, an agreement between Australia and Japan meant that the sample container was not allowed to be opened for inspection at the airport (Abe et al. 2011).

6.4 Security

The responsible for the security of the samples are the mission Investigators, which maintain the supervision of the samples when these should be analyzed with facilities outside of their laboratories. However, the Investigators should handle these samples in order to maximize the scientific yield of sample analysis (Hayabusa Sample Investigator's Guidebook)

6.5 Labelling and documentation

Marks and label give important information about a sample. Marks are number or codes which identify the specimen, whereas labels provide accessible information. Tha application on mark can occur in three stages: 1) after collection ; 2) after entering in the curation ; 3) when the sample is catalogued.

On stage 1 mark could consist of name of the site where the specimen has been sampled and a sequential number. On stage 2, an Entry Number can be assigned to the specimen, accompanied by a label giving information about the sample, e.g. origin, site, preliminary composition. Finally, on stage 3 the specimen Label could allow for recording on addition information with respect to stage 2, e.g. storage location, name of cataloguer, cataloguing date, name of curation, name of institution.

The following techniques are usually used to mark specimens (Brunton, 1984):

- Direct engraving or inscription, but this method is poorly suitable for small samples such as the extraterrestrial ones
- Associating to a paper tag

Labels should be the simplest as possible and should be prepared in order to be written in a permanent medium. Moreover, they should include the « history » of the sample, e.g. subjected processes, donations, exchange, etc.

7. Conclusions

Previous mission such as Hayabusa, Genesis and Stardust provide a basis to draw up a protocol for approach to and transport of a returned Mars sample. In particular the Hayabusa mission, being both the most recent, but also a non-US mission, offers much experience. However, the biohazard aspect of the transport and possible decontamination scenarios have only ever been developed by public health teams dealing with eg: high BSL viruses in Africa.

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In a worst-case scenario, samples from a damaged capsule may be recovered and immediately secured in a specialist container to go to the curation facility. This potentially could be done at the landing site with appropriate support. It will not be possible to use a portable receiving facility to analyse samples in any way. However it might well be used to make the sample safe, assess the seals and package it for transport to the curatorial facility.

8. Assumptions and dependencies

8.1 Assumptions

In order to move to WP6, certain assumptions need to be made about the requirements of the sample capsule and landing, including:

8.1.1 Landing site

All sample return missions have either performed on orbit capture (such as LDEF which was returned via the Shuttle) or landed in a hot sparsely populated desert area such as Utah or Woomera. In Zolensky and Sandford (2011), they state that they found the recovery using Woomera to be more robust than Utah. It is assumed here that the landing site will be on land, in a desert and probably at Woomera.

8.1.2 Temperature of the samples

Consideration needs to be given to the temperature of the capsule during recovery. The capsule will undergo the possible extreme temperatures of reentry (although protected by an ablative heat shield) and then land in a hot desert. It has been assumed here that rather than undergo repeated melt-freeze cycles, it would be preferable scientifically that the samples temperature be kept within room temperature range. If cold storage is required, then a subset of the samples could be sent to the vault storage facility which will have cold storage capacity.

8.1.3 Mass of Sample Return Capsule

The mass of the sample chamber is critical affects size of the Earth return capsule and size of the transport chamber and size of the curatorial facility. NASA's Mars2020 mission is currently being designed as the first stage of a MSR mission where a subsequent retrieval lander / spacecraft will collect its cached samples. (Obj C of the Mars2020 SDT Report). The estimate of sample size will be based on these mission requirements. Section 6.2.3.1 of the SDT defines a total sample mass of 500g divided over approximately 31 individual samples, ie : each one is 15-16g. It is also assumed that a sample may contain rock core, regolith, ice and gas. The argument for mass and size is important in appreciating what services might be deployed at the recovery site. For example, a field-deployable BSL-4 (based on a shipping container) would provide an invaluable facility in terms of assessing samples from the landing area etc. However, such a facility and its protected glove boxes is designed for small items (a culture plate for example).

8.2 Dependencies

The following questions will be put to the experts involved in the Genesis, Stardust, Hayabusa-1 and Osiris-REX recoveries. The work in WP6 is dependent on receiving answers to these questions.

<u>Questions to be put to experts:</u>

- What contingency scenarios did you plan for?

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- What field training did you carry out and how long did this take?
- How is the landing site dependent on size and mass of the capsule?
- Is current landing technology ie: parachutes suitable for Mars Return capsule?
- How do we prevent breakup of capsule on arrival?
- What environmental measurements did you make at the landing site?
- What procedures were carried out in the temporary cleanroom near the landing site ?
- With which equipment did you assess the state of the capsule?
- What security measures did you take to ensure the safety of the capsule?
- What type of container did you use to transport the capsule to the curatorial facility?
- How did you ensure no terrestrial contamination during the transport of the capsule?.
- What customs and regulatory arrangements were necessary for the transport of the capsule?
- Is there a regulatory specialist contact for the Australian side of the transport for Woomera?
- How is MSR capture being performed and how is the biohazard chain broken between Earth and Mars?

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