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D1.5 List of preliminary requirements – 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 EuroCares 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.

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, onlyanalogue 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 analogues	*Site*	*Test and calibration*	*Lander touchdown and rover mobility (Richter 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.

Brief overview of existing sample receiving facilites

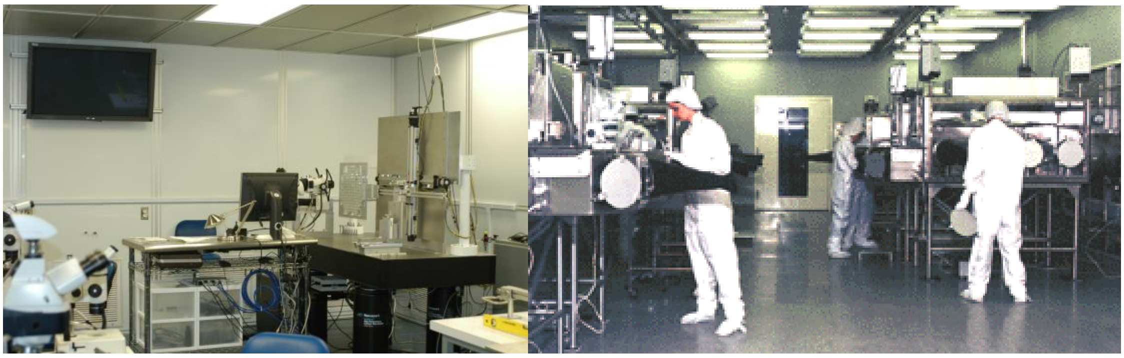
Planetary Material Sample Curation Facility of JAXA (PMSCF/JAXA):

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. ……..

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

A variety of extraterrestrial 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.

Stardust: 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 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…………

1- Samples required at for a curation facility

1.1 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 rocks	Picro-basalts	Mars (McSween et al, 2009)	Natural
	Basalts	Mars (McSween et al, 2009) The Moon Asteroids	Natural Natural 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 Impact melt rocks	The Moon Asteroids	Natural 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
	Mars permafrost	Mars (Smith et al., 2009)	Synthetic (Chevrier et al., 2007)
	Icy moons regolith	Titan	Synthetic
Meteorites	Chondrites	Asteroids	Natural
	Achondrites	Mars The Moon Asteroids	Natural Natural 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) Hydrothermal deposits (Callac et al., 2013 Salt deposits (Barbieri and Stivaletti, 2011) Carbonate mudmounds (Marlowe et al., 2014)	Mars	Natural

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) Meteorites (Blake et al., 2013) The Moon Asteroids	Raman spectroscopy
	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).

1.2 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 compound	Glycolaldehyde	Interstellar medium (Jorgensen et al., 2012)	GC-MS 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).

1.3 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).

1.4 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 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		
	??????		

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.

2- 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.

2.1 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.

4.2 Chemical samples

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

4.3 Biological samples

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

4.4 Technical properties samples

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

4.4 General requirement

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

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