

EURO-CARES

A PLAN FOR EUROPEAN CURATION OF RETURNED EXTRATERRESTRIAL MATERIALS



WORK PACKAGE 5

FINAL REPORT ON ANALOGUE CHARACTERISTICS

NECESSARY FOR THE CURATORIAL FACILITY

(DELIVERABLE D5.4)

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Preambule: WP5 Analogues in the context of the EURO-CARES project

The aim of the EURO-CARES project is to create a curatorial and analytical facility dedicated to extra-terrestrial samples brought to Earth from different bodies in the Solar System (Mars, plane-tary satellites, asteroids, the Moon), either by unmanned and/or by manned missions. These samples will require specific storage conditions and handling procedures during curation and analysis. Analogue samples will be crucial in evaluating and defining the protocols 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 will be necessary for the curation and analytical facility to have its own collection of well characterised analogue samples. Moreover, the analogue samples could be used in the testing of planetary protection measures, as well as validating new analytical methods.

Our approach to evaluate potential analogue samples needed assumptions about functions and activities expected from the curation facility. Limited to the very key points, and in agreement with reports from the other work packages, we defined the curation facility to have the following basic functions :

- opening of the returned sample canisters
- handling and preparation of the returned samples
- primary analysis of the returned samples, i.e. initial sample characterisation
- long-term storage of the returned samples

All these activities require analogue samples and materials at all stages starting from the intitial mission requirements to the final curation of returned samples.

Objectives of WP5

The **overall objective** of WP 5 is to create a **list of different types of samples** that would be required for a curation facility, and to create a preliminary list of analogue materials already available. These lists, including recommendations for the fabrication of new artificial analogues, have been completed over the course of the EURO-CARES project in response to the requirements established by the other work packages.

Detailed WP5 objectives

- To evaluate specific storage conditions and handling procedures during curation and analysis of extraterrestrial materials.
- To identify analogue samples crucial for evaluating and defining the protocols necessary to accomplish safe and sustainable handling of extra-terrestrial materials.
- To create a list of different analogue samples that would be required for a sample curation facility (e.g., analogues, standards, and witness plates).
- To create a preliminary list of analogues already available.
- To complete these lists over the course of this project in response to the requirements established by the other work packages.
- To include recommendations for the fabrication of new artificial analogues.

Brief overview of previous WP5 activity

To date, our activities have concerned:

D1.5 List of preliminary requirements – Analogue samples (Annex 1)

D5.1 A survey of the existing literature and the creation of a preliminary list of the kinds of samples necessary for a curation facility (including geological samples (rocks and minerals), chemical samples (ices and organics), and technical samples). A workshop to discuss the nature of analogues for a curatorial facility was held in Orléans February 4-5, 2016. The results of the literature survey and the workshop were presented (**Annex 2**).

D5.2. An expert workshop was held at the Senckenberg Gesellschaft für Naturforschung, Frankfurt, Germany in June $1-3^{rd}$. External experts with experience in the field of analogues were invited and discussions with them aided us to reach collective definitions of the various kinds of analogue materials needed, as well as a list of what are considered to be the most essential materials. The results of this meeting were presented as Deliverable 5.2 (**Annex 3**).

D.5.3. List of abstracts for oral and poster presentations at international conferences, seminars. The creation of templates listing the characteristics of chosen extraterrestrial-analogue rocks and minerals for the curation facility (**Annex 4**).

Definition of analogue samples useful for a curation facility

The samples brought back to the Earth will be very precious. Depending on mission target, returned samples will comprise a variety of materials and different sample types. Thus, it will be crucial to optimise sample preparation protocols, minimise the loss of matter, and avoid any contamination. It is for this reason that analogue samples and materials are important for testing many activities and procedures within the curation facility. Examples for such activities may be:

- > Develop and improve sample preparation and transportation protocols
- Elaborate optimized analysis protocols
- > Test and calibrate instrumentation
- > Aid data interpretation

We distinguish five different groups of **analogue samples**. The specifics of each group of analogue sample is given below.

Analogue sample	properties	purpose
Analogues	physical/chemical	analogues for extraterrestrial
		samples
Reference samples	physical/chemical; well characterised	for testing
Standards	homogenous, internationally recog-	for calibration
	nised physical/chemical	
Voucher specimen	duplicate materials that came poten-	assessment of potential con-
	tially in contact with extraterrestrial	tamination
	samples	
Witness plates	allowing for detection of, e.g., partic-	documentation of storage
	ulate, organic contaminants	conditions

Analogues are materials that have one or more physical or chemical properties similar to the Earth-returned extraterrestrial samples.

Reference samples are well-characterised materials with known physical and chemical properties used for testing and may not necessarily be the same materials as the analogues defined above.

Standards are internationally recognised, homogeneous materials with known physical and chemical properties that are used for calibration. They can also be used as reference samples in certain circumstances. They may be made of natural materials but are often produced artificially.

A **voucher specimen** is a duplicate of materials used at any stage during sample acquisition, storage, transport, treatment etc., e.g. spacecraft materials (including solar panels), lubricants, glues, gloves, saws, drills, and others. In addition, Earth landing site samples (from the touch down site) would be necessary in case of doubtful analysis, even if normally this type of contamination is not expected.

A witness plate is defined as material left in an area where work is being done to detect any biological, particulate, chemical, and/or organic contamination. It is a spatial and temporal document of what happens in the work area.

The following examples list some of the uses of the different types of analogues defined above:

- Sample handling (analogues with appropriate sample size and nature, and physical/chemical properties)
- Transport protocols (e.g. empty containers (reference samples), analogues with appropriate sample size and nature, and physical/chemical properties)
- Sample preparation protocols (analogues and reference samples exhibiting appropriate physical/chemical properties)
- Training of science (incl. external laboratories) and curation teams (e.g. reference samples, standards)
- > Long-term storage (e.g. witness plates, voucher specimen, etc.)

We note that for **curation**, two sets of analogue samples for training in non-sterile and sterile conditions should be made available for external laboratories and mission related hardware tests. The analogue samples should also be made available for public outreach associated with the curation facility, such as a museum.

For **storage within the curation facility**, on the other hand, analogue, reference samples, standard, and voucher specimens should be temporally and/or spatially isolated from extraterrestrial samples but they should be easily accessible. Witness plates, on the other hand, should be close to the returned extraterrestrial samples.

Types of analogue samples

The different groups of analogues samples should consist of a variety of types, e.g. grains, rocks, chemicals, etc. The types of an analogue sample may aid curation requiring special procedures. It will be determined by the nature of the returned extraterrestrial samples and will stand for a selected set of propoerties. In this context we distinguish:

1. **Material samples** are mostly analogues and reference samples that are particularly relevant for testing sample handling and preparation. They are analogue sample types with special properties, such as:

- Size (relevant for sample return of, e.g. drill cores)

- Porosity (relevant for sample return of e.g. pumice, obsidian)

- Density (relevant for sample return of mostly minerals, e.g. hematite, $d\sim 5$ g/cm³, gypsum 2,32 g/cm³)

- Roughness (e.g. Fontainbleau sandstone, ~270 μ m grains size (roughness ~1 μ m), obsidian roughness ~30 nm)

- Consistency (e.g. soft clay (unconfined compressive strength $<25~q_u(kPa)$, hard clay unconfined compressive strength $>\!400~q_u(kPa))$

- Physical properties (ice)

2. **Geological samples** may be naturally occuring rocks (volcanic (extrusive and plutonic), sedimentary, hydrothermal, impactites etc.), minerals known to occur on extraterrestrial bodies, or even artificially produced rocks, e.g. a Mars-like basalt (Fig. 1). Some of these should also contain natural microbial fossils with organic signatures. Geological samples are natural mixtures of various components that consist of one or more minerals or rock fragments.



Figure 1. Types of geological samples

3. Chemical samples comprise both "known" and "unknown" molecules (Fig 2). "Known" molecules include, for instance, those detected in the interstellar medium, in meteorites, or in comets. There is a wide range of simple to complex molecules occurring in the interstellar medium, including fullerenes with over 60 carbon atoms. Over a thousand organic molecules have been detected in carbonaceous meteorites and micrometeorites, while the recent Rosetta mission to comet 67P/Churyumov–Gerasimenko detected both volatile and high molecular weight molecules (Fray *et al.* 2016, Altwegg *et al.*, 2016; Goesmann *et al.* 2015).

"Unknown" molecules are those that could potentially occur in extraterrestrial bodies, such as comets, or in the atmospheres of other planets and satellites, such as the tholins believed to form in Titan's atmosphere. They are also represented by insoluble matter in meteorites. Other unknown samples would be materials artifically-doped with organics and/or fossil microorganisms.



Figure 2. Types of chemical samples

Recommendations

- Start the analogue collection now! Make sure that the collection (including witness plates) is ready early enough for protocol testing and even during the preparatory phase of a mission or the building phase of a curation facility.
- > Curation should accompany mission planning and development from the start.
- At the start of a mission, the science team should make recommendations regarding suitable standards to be used in the curation facility.
- > Use the analogues for space mission instrument validation.
- Make sure there is enough material. Degree of homogeneity will depend upon the use of the analogue.
- The analogues need to include simple to complex materials to demonstrate that the endto-end workflow is flawless.
- However, specific analogues will be needed for specific processes. It will not be necessary that one analogue serves the whole end-to-end flow.

List of samples deemed necessary for a curation facility:

1. Natural analogues

Rocks

Primitive basalt Anorthosite Dolerite Tuff Suevite Mudstone Sandstone Lunar regolith Chondrite (CC, OC) HED meteorites Other meteorites, .e.g, iron meteorites, achondrites Rocks containing natural microbial (and carbonaceous) fossils

Minerals

Olivine Pyroxene Plagioclase Metal (Fe/Ni) Jarosite Magnetite Hematite Calcite Dolomite Gypsum Anhydrite Sulphides (troilite or pyrrhotite) Mg smectites Serpentine Ices Silica (Opal)

Gas

 13 CO₂ (to be obtained on demand – not archived), 13 CH₄ (if needed)

Liquid

If needed

2. Manufactured analogues

Regolith/soil Soil mixtures (e.g. with perchlorate, ice) Icy/dusty mixtures Samples doped with organic matter (cometary/asteroid simulants) Samples doped with microbial fossils (Mars simulants)

Manufactured analogues include mixtures of different components, biologically-doped samples, and organically-doped samples. Note that it is essential that the source provider maintains quality control. Moreover, manufactured analogues will be produced on a case by case basis.

Another important point to keep in mind is that the manufactured analogues need to be produced in sufficient amounts to ensure uniformity between batches.

Spreadsheets and database

All the data related to analogue samples are stored at this website address encoded in html/php and MySQL:

http://www.euro-cares.eu/wp5/database/database.php



The data is presented in the form of spreadsheets for each type of sample, an example of which is presented below:

🥝 Calo	ite from unk	Reference: EURO-CARES-C4
Target Bodies: X Mars Moon Asteroids Other() Target Geological		Photo credita:
Context:		albridges b. or escusion
Type of Sample	X Analogue Reference sample Standard for: Voucher specimen Witness plate	
Nature	X Rock X Mineral Gas Liquid Synthetic Amorphous material	
General Geological Description	Petrography: Fine grained carbonate mud Mineralogy (for rock samples): XRD on powde Mineral type (for mineral sample): Carbonate Chemistry: CaCO3	er pellets, near pure CaCO3
Physical properties	Cohesion: hand sample Density: 2.7 Hardness: 3 (Mohs) Porosity: TBD (%) Mass: 100 (g) Volume: TBD (ml) Health hazard: no Any other relevant physical properties data:	
Source	GPS:	
Collector	Name: Address:	
Links to other WPs		
Further comments, Information		
Associated data		
History of the sample		

The database has a search engine based on parameters, such as sample name, reference number, type and nature of samples, or their physical properties, as shown below:

Anour	121 BY21 B		
ABOUT	Search for samples:		
WP5 Scientific team	Name:		
Sample types EURO-CARES	*		
1071952822	Reference number:		
REGISTER	B		
Login :	Target body:		
Password :	1		
Loge	Type and nature of samples		
To ask for registration	Туре: 1		
please fill the following form:	If standard, for which instrument: +		
Ask for registration	Nature: 2		
	Physical properties		
	Cohesion: 1		
	Density. Between and		
	Hardness/compressive strength: Between a z Mohs and a z Mohs		
	Werks seem: 1 (both, 2 (general), 2 (method, 2 (framely), 2 (interfed), 2 (otherware thirtype), 7 (spraw), 8 (spraw), 9 (
	Porcenty between 0 I to and 100 I to		
	Mass nigher than: g		
	Volume higher than: ml		
	esternt mout		

Finally, registered users can access the administration panel allowing them to add, modify or delete samples from the database. This interface permits homogenisation of the data (automatic nomenclature, drop-down lists, examples, pre-filled boxes...) and to avoid mistakes by data verification before validation.

REFERENCES

K. Altwegg, et al., 2016. Prebiotic chemicals-amino acid and phosphorus-in the coma of comet 67P/Churyumov-Gerasimenko.Sci. Adv. 2(5):e1600285. doi: 10.1126/sciadv.1600285.

N. Fray et al., 2016. High-molecular-weight organic matter in the particles of comet 67P/Churyumov–Gerasimenko, Nature, 538(7623), 72-74, 10.1038/nature19320, 2016

F. Goesmann F., et al., 2015. Organic compounds on comet 67P/Churyumov-Gerasimenko revealed by COSAC mass spectrometry, Science, 349, 6247, 10.1126/science.aab0689.

Annex 1. D1.5 List of preliminary requirements – Analogue sample General background comments

Analogue samples have been used in space exploration for testing instruments for *in situ* missions to help interpretation during a mission or to simulate a particular phenomenon. It is important to note that analogue samples are not calibration samples. Indeed, most of the time the teams in charge of instrument development use calibration samples that are not relevant of the extraterrestrial body being studied but, on the contrary, are particularly well-suited for a given technique or instrument. For example, silicon is used to calibrate Raman spectrometers because it is associated with a very high signal/noise ratio and because its Raman spectrum is very simple (one very strong peak). Natural analogue samples are generally very complex and require the use of several instruments to be characterized. Thus, analogues such as rocks and minerals are more pertinent for facilitating identification and interpretation of particular processes of formation of a rock or mineral. Nevertheless, natural analogues are extremely useful for testing the complementarity of different instruments of a payload (e.g. Bost et al. 2015).

During a previous workshop organized by the CNES in December 2013, "analogue" types were sorted as follows (Fig. 1):

- The natural analogues are mainly geological samples. They range from particular landscapes or formations on Earth that can be used to test rovers (e.g. AMASE) or that are relevant for their geology (e.g. Rio Tinto), to hand specimens of rocks pertinent for their petrology (e.g. basalt from Tenerif or meteorites). Several reports and articles still exist on analogue sites or analogue rocks (e.g. Preston *et al.*, Bost *et al.*). Some places are also relevant for their microbial biotope potentially representative of potential extraterrestrial metabolism (e.g. Yellowstone).
- The simulated analogues are mainly interesting for chemistry. Simulation chambers are used to mimic the environmental conditions on a particular body or in the interstellar medium and to synthetize analogue molecules (d'Hendecourt *et al.*, Danger *et al.*, Meinert *et al.*...). Some artificial rocks have also been synthetized (Bost *et al.*...). Finally, cultures of microorganisms in laboratory can be used to understand particular metabolic pathways. These analogues samples are generally very low quantity and are thus difficult to use for the test of space instrumentation.
- The numerical analogues are obtained by computational simulations.



Figure 1. Classification of analogue types and uses

The EURO-CARES project focusses on all processes related to the curation and analysis of samples brought back to Earth from different bodies in the solar systems (Mars, asteroids, the Moon...). The analogue requirements for a sample return mission are different to those for an *in situ* mission. For example, whereas relatively large samples are used for payload testing, the small quantity of precious materials brought back to Earth (a few grams per sample) mean that all curation facility procedures need to be tested on samples of equivalent small size. Moreover, for planetary protection considerations, analyses of the samples will need to be made in particular clean-room conditions of biosafety level. For practical reasons, and due to the small amount of samples required, it is thus obvious that **the analogue samples must be stored in a dedicated collection located in the future EURO-CARES facility**.

In this first review and prospective of available documents, we describe the interest of the future analogues stored in the EURO-CARES facility. In this view, we have categorised the curation facility analogues in three types:

- (1) natural and synthesized scientific analogues,
- (2) calibration analogues, and
- (3) contamination analogues.

1- Interest of analogue samples in the curation facility

The curation facility will contain several analytical instruments dedicated to chemistry, mineralogy and biology. The requirements for the facility infrastructure and the pertinent techniques will be described in the framework of tasks 1.3 and 1.4. Depending on the chosen techniques, different analogues samples will have to be considered. The analogue samples will be used for:

- Training on sample manipulation.
- Training on sample preparation.
- Configuration and optimisation of instrument setup.
- Controlling measurements.
- Calibrating the instruments.
- Searching for possible contamination.

These considerations should also be taken into account when selecting samples during a mission.

1.1- Sample manipulation

It is probable that different instruments will be interfaced together in a controlled atmosphere chamber. The samples will have to be manipulated to pass from one technique to another. Depending on their size, weight or hardness, this manipulation phase could be particularly critical and it is thus important for previous training.

1.2- Sample preparation

Some techniques will require sample preparation (cutting, polishing, crushing...). Due to the value of the returned samples, it will be very important to train before on preparing analogue samples to limit the loss of material during this phase.

1.3- Instrument setup

The analogue samples will be useful to establish protocols for sample analysis. The aim here will be to carry out the different analyses on the returned sample in a logical way, permitting a maximum of information to be obtained with the minimum amount of material.

1.4- Controlling measurements

One of the main interests of analogue samples will be to help interpretation of the analyses. These samples can be used as control after, but also before, the measurements via analytical databases.

1.5- Calibration

Even if calibration samples are not analogue samples *sensu stricto*, in the case of the curation facility project, since this kind of samples will be dedicated to the instruments of the facility it is important to add them to the analogue samples collection.

1.6- Contamination

All measurements made on the returned samples will have important scientific impact. It is thus crucial to be able to avoid any contamination. Analogue samples of possible contamination must thus be considered in the collection.

1.7- Help for in situ missions

The techniques and methods developed and improved during instrument calibration at the curation facility could be very useful for determining the minimum amount of sample necessary to take during the mission and, thus, help sample selection.

In the following, we consider the possible types of samples that could be relevant for the above activities based on the three sample category types noted above, natural and synthetized scientific analogues, calibration analogues, and contamination analogues.

2- Natural and synthetic scientific analogues

2.1- Geological samples

(1) Existing, previously characterised samples, e.g. in an existing collection (e.g. the International Space Analaogue Rockstore, ISAR; <u>www.isar.cnrs-orleans.fr</u>)

- Volcanic rocks, cumulates, volcanic sediments, various types of meteorites, evaporitic sediments, hydrothermal sediments, minerals...

(2) Newly aquired rock and mineral samples (depending on the outcomes of our study)

(3) Artificial samples, e.g. chemically-synthesised minerals or rocks (cf. artificial martian basalt, Bost *et al.*, 2011; basaltic glass calibration samples, Favre *et al.*, 2011). Many of such samples were created for the Mars rovers.

2.2- Biological samples

(1) Living organisms that could potentially exist in the returned rock samples – of extraterrestrial origin or terrestrial contamination. Analogues in this case would consist of strains that have been acquired from a natural analogue environment or strains that have been grown in specific analogue conditions.

(2) Analogues of microfossils, *i.e.* analogue rocks containing the kinds of microorganisms that could have been fossilised on another planet (Mars), or artificially fossilised strains of these kinds of microorganisms.

2.3- Chemical samples

(1) Samples of natural, extracted or synthesised abiotic/prebiotic molecules, such as amino acids, peptides, proto-sugars, ...etc.

(2) Samples of biological organic molecules, either natural, extracted or synthesised (?), such as lipids, sugars, amino acids, nucleic bases etc.

3- Calibration analogues

The types of analogues needed for calibration will be technique specific, e.g. Dyer et al., 2015

3.1- Mineral and material samples

Example for Raman: silicon, quartz...

3.2- Chemical samples

As above in 2.3?

3.3- Other

Calibration target

4- Contamination analogues

4.1- Material samples

Ex: Resin used for the rover..., other materials used in instrument construction

4.2- Chemical samples

Molecules desorbed by the resin or other materials used...

4.3- Biological samples

Most common prokaryotes and viruses.

References:

Bost, N., Westall, F., Gaillard, F., Ramboz, C., and Foucher, F., 2012. Synthesis of a spinifextextured Basalt as an analog to Gusev crater basalts, Mars. Meteoritics & Planetary Science 47, pp. 820-831. doi:10.1111/j.1945-5100.2012.01355.x.

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M. D. Dyar et al., 2015. Calibration suite for Mars-analog laser-induced spectroscopy. LPSC abstract #1510.

C.Fabre,S.Maurice,A.Cousin,R.C.Wiens,O.Forni,V.Sautter,D.Guillaume, 2011. Onboard calibration igneous targets for the Mars Science Laboratory Curiosity rover and the chemistry camera laser induced breakdown spectroscopy instrument, Spectrochim. Acta Part B 66 (2011) 280 – 289.

D. Vaniman, M.D. Dyar, R.C. Wiens, A. Ollila, N. Lanza, J. Lasue, M. Rhodes, S.M. Clegg, 2012. Ceramic ChemCam calibration targets on Mars Science Laboratory, Space Sci. Rev. 170 (2012) 229–255

Wiens, R., et al., 2013. Pre-flight calibration and initial data processing for the ChemCam laserinduced breakdown spectroscopy instrument on the Mars Science Laboratory rover. Spectrochimica Acta Part B 82 (2013) 1–27

Annex 2: Expert Workshop: Analogue Sample Characteristics, February 4-5, 2016, Orléans, France

Programme:

Thursday: 9h-18h

9.00-9.15 Brief introduction to workshop objectives

9.15-12.30 Discussion as to which kinds of samples would be absolutely essential in a curation center

(as opposed to collections of analogue samples useful for specific studies)

12.30-13.30 Lunch (provided)

13.30-17.30 Discussion of the characteristics of the selected rock types

1930 Dinner at a local restaurant (paid for by EuroCares)

Friday: 9h-16h

9-12.30 Continuation of discussion of the characteristics of the selected rock types

12.30-13.30 Lunch (provided)

13.30-16.00 Conclusion and report writing

Participants:

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Results:

The participants of the WP5 workshop came to the conclusion that a selection of 8 rocks and 15 minerals would be sufficient for the purposes of the Curation Facility. These rocks and minerals were chosen because of their pertinence for :

- Composition/textural similarities with planetary materials;
- Relevance for sample handling, processing, training
- Contaminants

The selected rocks include: primitive basalt, OIB (Oceanic Island Basalt), anorthosite, dolerite, tuff, suevite (impact breccias), mudstone, sandstone.

The selected minerals include: jarosite, goethite, hematite, Ca-carbonate, Fe-carbonate, Mg-carbonate, gypsum, anhydrite, perchlorates, sulphides, Mg-smectites, illite, chlorite, silica (opal, quartz and tridymite), ices.

N.B. Generic dust/regolith to be made up of mixtures of powdered basalt and minerals as needed (with addition of H_2O ice for SPA Basin on the Moon).

Powdered (not only powdered, maybe also some with chondrules that will pop out and roll around) meteorite should also be considered to represent asteroidal material (N.B. rare therefore to be used only in specific cases).

For each rock and mineral listed above, a data sheet listing the characteristics is being drawn up, following the example shown in below. This work is ongoing and should be completed by the end of April.



In the coming months, the following tasks need to be addressed:

- Complete list (also with biological and chemical samples)

- Choose best analogues of each category/type of rock at next meeting (1-2 June, Frankfurt am Main)

- Implicate outside specialists.
- Iterate list with WP 4, and 2
- Write the final report

Annex 3: List of presentation abstracts

Conference	Title	Authors	Oral/Poster
Missions to Habitable Worlds,	Analogues for planetary	Frédéric Foucher, Frances	Oral
October 2015	1115510115	Bost et al.	
D			
Designing a European extra- terrestrial sample curation	Storage and usage of ana- logue samples in an extra-	J. Ziptel, F. Westall, F. Foucher and the FURO-CARES Consorti-	Oral
facility. Vienna, Austria, 14-15	terrestrial sample curation	um	
April 2016	facility		
EURO-CARES WP5 workshop,			
2016			
EANA (European Astrobiology	EURO-CARES: European	Caroline Smith, Sara Rus-	Oral
network Association), Greece, 27-29 Sentember 2016	Curation of Astromaterials Returned from Exploration	sell, Frances Westall and the FURO-CARES team	
	of Space		
EANA (European Astrobiology	Reflections on the definition	Frances Westall, Jutta Zipfel,	Oral
network Association), Greece, 27-29 September 2016	of analogues and conse-	Frederic Foucher, et al.	
	CARES project		
European Commission Earth	Analogues in EURO-CARES	J. Zipfel and WP5	Oral
Analogue Workshop, REA, Brussels Belgium			
12/10/2016			
EURO-CARES WP4 workshop,	EURO-CARES WP5: Ana-	F. Westall, J. Zipfel, F. Foucher,	Oral
Paris, France, 13 October	logues and instrumentation	J. Bridges, V. Debaille, L. Folco, I. Michalski, P. Woznikiewicz, I.	
2010		Martines-Frias, K. Joy, M. Lee,	
		J.R. Brucato, M. Viso, N. Bost, A.	
		Hutzler, G. Kminek, H.	
		Zolensky, C. Smith, O. Bacon, M.	
		Van Ginneken & L. Ferrière	
Journées de la SFE, Lyon,	SMITH Caroline, Foucher, F.	EURO-CARES	Poster
2016	CARES		
48th Lunar and Planetary	ANALOGUE SAMPLES IN AN	J. Zipfel, F. Westall, F. Foucher	Poster
Science Conference, The	EUROPEAN SAMPLE	and the EURO-CARES Consorti-	
2017 woodiands, warch 20-24th,	FURO-CARES PROJECT	um	
AbSciCon, Phoenix, 24-28	GENERAL REFLECTIONS ON	F. Foucher1, F. Westall1, J.	Oral
April 2017	THE DEFINITION OF	Zipfel2, N. Bost1 and the EURO-	
	ANALOGUES AND	CARES Team	
	EURO-CARES PROJECT		
AbSciCon, Phoenix, 24-28	ANALOGUE SAMPLES IN A	F. Westall, J. Zipfel, F. Foucher	Oral
April 2017	EUROPEAN SAMPLE	and the EURO-CARES Team	
	EURO-CARES PROJECT		

14th International Planetary Probe Workshop ESA The	European curation of as-	C. Smith, S. Russell, F. Foucher	Oral
Hague, 12-16 June 2017	exploration of space: the		
	EURO-CARES pro-ject.		
14th International Planetary	General reflections on the	F. Foucher, F. Westall, J. Zipfel,	Oral
Probe Workshop, ESA, The	definition of analogues.	N. Bost and the EURO-CARES	
Hague, 12-16 June 2017		Team	
ISSOL San Diego 16-21 July	Life on Mars: returned sam-	F. Westall, J. Zipfel, F. Foucher,	Poster
2017	ples and their storage, the	C. Smith, S. Russell, K. Hickman-	
	EURO-CARES project	Lewis, M. Viso	
EANA Aarhus, Denmark 14-17	Analogues for a European	F. Westall, J. Zipfel, F. Foucher,	Poster
August 2017	Extraterrestrial Sample	C. Smith, V. Debaille, L. Folco, J.	
	Curation Facility	Bridges	

Annex 4: EURO-CARES Workpackage 5 expert workshop, June 1-3 2016, Senckenberg Forschungsgemeinschaft, Frankfurt

Participants

Frances Westall (CNRS-Orléans), Jutta Zipfel (Senckenberg), Frédéric Foucher (CNRS-Orléans), Oliver Bacon (NHM London), Nicolas Bost (CNRS-Orléans), John Bridges (Univ. Leicester), Vinciane Debaille (Univ. Libre Bruxelles), Ludovic Ferrière (NHM-Vienna), Luigi Folco (Univ. Pisa), Aurore Hutzler (NHM-Vienna), Gerhard Kminek (ESA-ESTEC), Hilde Schroeven Deceuininck (ESA-Harwell), Caroline Smith (NHM London), Matthias van Ginneken (Univ. Libre Bruxelles), Michel Viso (CNES), Hajime Yano (by skype) (JAXA), Michael Zolensky (NASA-JSC).

The external experts invited to this workshop are:

• Nicolas Bost, CNRS-Orléans, created the International Space Analogue Rockstore (ISAR; www.isar.cnrs-orleans.fr)

• Gerhard Kminek, ESA, Planetary Protection officer

• Hilde Schroeven-Decuininck, ESA, responsible for the ESA analogue sample facility at Harwell, UK

- Michel Viso, CNES, responsible for Astrobiology and Planetary Protection
- Hajime Yano, ISAS/JAXA, Hyabusa sample curator
- Michael Zolensky, NASA-JSC, cosmic dust curator

Workshop Progamme

June 1st

Smith/Westall/Zipfel: Introduction to the EURO-CARES project, WP5 (analogue) activities and general logistical information

Presentations from the experts:

Hilde Schroeven-Decuininck on ESA activities regarding analogue samples Frederic Foucher on analogue samples from the engineering point of view Nicolas Bost on the ISAR Mike Zolensky on curation of cosmic dust Gerhard Kminek on planetary protection issues Michel Viso on shared curation facilities

June 2nd

Report on WP 2 workshop by Luigi Folco Hajime Yano on Hyabusa curation Discussion: Glossary definitions What analogues for what purpose? Storage considerations for WP3 Data base Evaluation of minerals and rocks already chosen

June 3rd

Agree on terminology Make recommendations for:

- function and material types
- level of detail

Brief summary of expert presentations:

1. Hilde Schroeven Decuininck: ESA analogue sample curation facility

The activity supported by ESA at the Harwell site near Oxford concerns the curation of analogue materials for missions and not the curation of extraterrestrial samples per se. It takes place within the programmatic framework of missions (MREP, EXPERT etc.) involving the ISS, the Moon, and Mars. It involves, for example, the collection and curation of samples (both natural and synthetic) from dust to pebble size, of different types (soil/regolith, frozen, volatile) for preparing sample return missions from the engineering point of view, i.e. physical/geotechnical properties, chemical/mineralogical properties, and long-term sustainability.

The objectives of these activities are to:

- build an analogue sample curation facility
- build up central knowledge and expertise
- enhance central collection by achieving reference standards.

The analogue sample curation facility will contain basic tools for characterising the materials but, if more in depth knowledge is necessary, laboratories outside the centre will be called upon.

2. Frédéric Foucher: analogues from an engineering point of view

Analogue samples can be classed into those useful for geological (rocks, minerals), chemical (organic molecules), or biological (extant/fossil life) purposes. In an extraterrestrial sample curation facility, samples are also necessary for testing sample handling, preparation, and storage. The latter implicates the use of representative samples of different nature (solid, liquid, gas), size, porosity, density etc.

The type of analogue samples used for tests will depend on the mission objective. For instance, while samples containing extant traces of life may be relevant for Mars or the icy satellites, they are not relevant for the Moon.

The aspect of materials coupons to control potential contamination throughout the extraterrestrial sample handing process is important.

3. Nicolas Bost: the International Space Analogue Rockstore (ISAR)

The objective of the ISAR is to create a collection of Mars-analogue rocks and minerals that have been well-characterised. Together with a fully-detailed, web-based and readily available database, the ISAR collection is used for scientific testing of mission payloads, e.g. ExoMars, and for related science activities (Bost et al., 2012, 2013, 2015).

This collection is representative but non-exhaustive. It is in the process of being transferred to the ESA curation facility for analogue samples.

4. Mike Zolensky: Cosmic dust curation at the NASA-Johnson Space center, Houston

The Stardust mission provided an excellent platform for learning about the handling, preparation and curation of small-sized samples, such as cosmic dust. The unfortunate crash of the return capsule on landing emphasised the need to be prepared for all eventualities, both worst case scenarios, as well as obtaining more material than envisaged.

The building of the Stardust curation facility demonstrated the usefulness of a modular system in which also different location in one clean room may have different levels of cleanliness.

An important lesson learnt was the necessity to use witness coupons at all stages of activities.

Regarding analogues of cometary dust, after the Stardust mission it became clear that the previously used cosmic dust collected in the stratosphere is largely terrestrial contamination and not a particularly good analogue for cometary dust.

5. Gerhard Kminek: Planetary protection

Suitable planetary protection measures need to be emplaced for sample return missions that are driven by the search for traces of extraterrestrial life, such as Phobos/Deimos, Mars or the outer icy satellites. This requires the preparation of reproducible standard samples (natural or synthet-ic), in sufficient quantity, of natural and artificial materials. These samples are used for testing sample preparation techniques, such as organic extractions.

These samples are also used for experimental testing of the inactivation of microbial cells by heat and radiation methods, as well as the survival of microorganisms in rocky materials subjected to experimental impact studies.

It will be necessary to identify laboratories to run tests on natural and synthetic analogue samples.

Given the lead times involved, it is important to start preparing for sample return NOW. The planetary protocols are detailed in Allwood et al. (2013) and Kminek et al. (2014)

6. Michel Viso: Facility or facilities

Extraterrestrial sample return should be viewed in the context of facilitIES and not a single facilitY. While the receiving facility will probably be in the US, the curation, transient quarantine and analytical facilities will be distributed around the world.

Sample curation and the distribution strategy, managed by an international body, should be established in the framework of participating, global partners.

It is recommended that there be no backward track for samples in order to prevent contamination and that the curation facilities should consist of independent modules to avoid single point failure. These modules could be adjacent to each other or spread across different countries, including within Europe.

Finally, seeing as sample return from Mars will present the greatest challenge, it is politic to prepare now.

June 2nd

7. Luigi Folco: Report from WP2 (Planetary Protection)

WP2 reviews planetary protection policies at a national and international levels, with a definition of methods, protocols etc, and provides recommendations with respect to forward and backward contamination, biohazard detection, and security.

8. Hajime Yano: Curation for Hyabusa samples

The Hayabusa mission activities were described from the conception of the mission to the distribution of samples. Importantly, the receiving facility was designed in parallel with the design of space craft and sampling device. Practice in opening the return canister opening took one year! The awarding of Hyabusa samples was dependent upon demonstration of scientific excellence by the competing labs. For this, a blind sample testing was undertaken, using as analogue samples the carbonaceous chondrite Allende and the ordinary chondrite Valdizzina (Yano et al., 2003). Curation facilities for the return of the Tanpopo mission on the International Space Station consist of a modular structure with different levels of cleanliness.

Of note, the budget for constructing the receiving facility was only received after the successful launch of the mission.

Discussion points

- 1. Glossary:
- Sample receiving facility

In this report, we assume that there is a SR facility (probably in the US) and that it is being addressed by other WPs. Obviously flight spare hardware/engineering models will be needed for staff training (but these are not classed as analogues from our point of view)

- Curation facility has the following functions

- opening of the returned sample canisters
- handling and preparation of the returned samples
- basic analysis of the returned samples, i.e. initial sample characterisation
- long-term storage of the returned sample

Sample types:

- Analogues are materials (e.g. rocks, minerals, ices, gases) that have one or more physical or chemical properties similar to those expected in extraterrestrial samples returned with SR missions.

- Reference samples are well-characterised materials with known physical/chemical properties used for testing the flow of the whole or part of the process. They may not necessarily the same material as the analogues defined above.

- Standards are internationally recognised, homogeneous materials with known physical/chemical properties that are used for calibration (e.g. silicon for Raman spectrometry). They can also be used as a reference sample in certain circumstances. They may be made of natural materials (e.g. the Belemnitella americana from Pee Dee Formation in South Carolina, used for 13C isotope studies) but are often fabricated artificially (e.g. the calibration targets used on the instrument ChemCam in the mSL mission).

- A voucher specimen is a duplicate of materials used at any stage during sample acquisition, storage, transport, treatment etc, e.g. space craft materials (including solar panels), lubricants, glues, gloves, saws, drills....., and stored for when needed.

Finally, Earth landing site samples (touch down site) could be necessary in case of doubtful analysis even if normally this type of contamination is not excepted.

- A witness plate is defined material left in an area where work is being done or assessed for e.g. biological, particulate, chemical, and/or organic contamination. It is a spatial and temporal document of what happens in the work area.

In order to illustrate the different types of samples, the example is given for a camera in Fig. 1.



Figure 1. Illustration of the different type of samples taken into consideration by the WP5 of the EURO-CARES project. The example is given for a standard camera. The analogue here is a volcanic bomb representative of expected structures on Mars, the witness plate is a sheet of paper that will stay in the area where the observation are made, the voucher specimen is the carrying bag of the camera that can potentially appear on an image, the reference sample is a pen that is useful to have an idea of the scale, and the standard is a ColorChecker target used to calibrate cameras.

2. Analogue materials:

- State of matter - analogue materials may have different states e.g.:

- solids including ices
- liquids
- gases

- Type – analogues may be natural or manufactured

Natural analogues include:

- rocks (all size ranges, including dust)
- minerals
- regolith

N.B. these materials could contain biological (extant and/or extinct) and/or organic components.

Manufactured analogues include:

- mixtures of different components in predetermined ratios for specific purposes
- biological doped samples
- organically doped samples

N.B. manufactures analogues need to be produced in sufficient amounts such as to ensure the uniformity of each batch. The source provider needs to maintain quality control between batches. Such samples will be produced on a case by case basis.

3. What analogues for what purpose?

- Sample handling requires samples with different physical or geotechnical properties, e.g. porosity, yield strength, compressive strength, density, temperature, cohesivity; as well as electrical and magnetic properties; different size distributions; and in different states of matter (solid/liquid/gas).

For example, analogues for returned samples from:

e.g. Mars could be an homogenous basalt, a mudstone, coarser grained (sand to ?conglomerate?) sediment, soil, dust;

e.g. for asteroid could be meteoritic material, loose regolith-like material, and dust;

e.g. Moon could be lunar samples, regolith, icy samples.

N.B. It will be important to produce protocols for keeping the analogues samples temporally and/or spatially isolated from the returned extraterrestrial samples. This does not necessarily mean that they need to be in the same room.

- Transport protocols for movement of the returned samples within the facility and for shipment out of it. It will be necessary to practice with empty containers and appropriate analogue samples (cores, fragments, dust) (sample size and nature are important). In this case, analogue samples exhibiting different physico-chemical-technical etc. properties will be necessary.

- Sample preparation protocols, for example, sectioning, powdering, splitting, chemical/heat extraction, and imaging (optical-SEM EDS). All analogue types exhibiting appropriate physical/chemical properties will be appropriate.

- Training of science and curation teams and science lab quality assessment, *i.e.* making sure that the external laboratory facility can handle/analyse the returned samples. ISAS/JAXA did a blind test of laboratories interested in analysing the Hyabusa 1 samples (Kushiro *et al.*, 2003). Such activities would use reference analogue materials.

- Long term storage needs to be tested using witness plates, hardware samples, voucher specimen and reference materials (including frozen materials).

4. What mass?

The mass necessary will depend on the objective and analogue types. Suggested masses will be driven by mission architecture and the target body of interest, as well as the defined science requirements, and availability.

We suggest the following:

~40 kg of terrestrial analogues (rocks)

~1 kg minerals

- ~1 kg meteorites
- 5. Storage considerations for WP3:

Analogue, reference, standard, and voucher samples all need to be isolated from each other and temporally and/or spatially isolated from extraterrestrial samples while, at the same time, being accessible.

N.B. In the case of restricted Earth-return samples (e.g. from Mars or Europa), the above listed materials should strictly NOT share space with the extraterrestrial samples either temporally and/or spatially.

With respect to witness plates, those in use actively need to be in close proximity to the returned extraterrestrial samples, while the past plates need to be stored elsewhere.

N.B. Since planetary protection aspects need to be addressed from the beginning of the curation activities, contamination assessment and control plans should be emplaced during mission planning and development.

6. Data base

There should be one database for the curatorial facility in which different database subsections, e.g. the analogue database, will be embedded.

7. Evaluation of minerals and rocks already chosen

N.B. Standards and reference materials will be chosen in accordance with WP2, 3, 4

The following is a suggested list of analogue samples, the selection of which will be mission dependant:

Natura	l analogues				
Rocks	Primitive basal	lt	Minerals	Olivine	
	Anorthosite		Pyroxene		
	Dolerite		Plagioclase		
	Tuff	Metal (Fe/Ni)		
	Suevite,		Jarosite		
	Mudstone		Magnetite		
	Sandstone		Hematite		
	Lunar regolith		Calcite		
	Chondrite (CC	,OC)	Dol	omite	
	HED meteorites		Gypsum		
Anhydrite Perchlorates					
	Sulphides (troilite/pyrrhotite)				
	Mg smectites				
		Serpentine			
		Silica (amorphous/opal)			
		Ices			

Gas* 13CO2

13CH4

Liquid* * Provided on demand

Manufactured analogues Regolith/soil Soil mixtures (e.g. with perchlorate, ice) Icy/dusty mixtures Doped samples (biological) Doped samples (organic)

8. Analogue sample spreadsheets

Each spreadsheet for the proposed analogue samples (Figure 2) contains information regarding its:

- nature and provenance
- EURO-CARES code number
- the target extraterrestrial body for which it is an analogue (specific mission, if relevant)
- the target body geological context
- the curation facility storage
- the analogue's state of matter
- a general geological description of the target including petrography, mineralogy, chemistry..

- physical properties, including density, hardness/compressive strength, porosity, tenacity, cleavage, fracture: electrical properties, magnetic properites, thermal behaviour

- health risks
- location of the sample, if relevant
- other information
- associated data
- history of the sample

The spreadsheet data for each analogue will be an integral part of the database.



Figure 2. Template of the analogue spreadsheet data.

Add an example of an analogue rock and mineral. Standard TBD.

9. WP 5 Recommendations:

- At definition of a sample return mission, the science team can make recommendations with respect to suitable standards to be used in the curation facility (cf. WP4)...

- Finally, Earth landing site samples (touch down site) could be necessary in case of doubtful analysis even if normally this type of contamination is not excepted.

- A second set of analogue materials is necessary for training in non-sterile conditions (except for biohazard testing)

- A third collection will be needed for training in sterile conditions (Action item: ask the PP group what they need)

- Other analogue samples should be made available for public outreach associated with the curation facility, such as a small museum.