WP1: Knowledge Capture and Requirements Review

Preliminary report - WP3: Facilities and infrastructure

We summarize here the current knowledge on "facilities and infrastructure" (i.e., state of the art 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 tried to cover 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 (including the construction, operation, security, etc.) to the storage of the samples (monitoring, sample holder, etc.) as well as the curation (sample handling and database).
For 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. We also used 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.

Building design (including the construction, operation, security, etc.)

The infrastructure should be designed and constructed in respect to prevent sample contamination and alteration on one hand, and to prevent potential biohazards from the sample on the other hand. All should be done to avoid not only terrestrial particles and organisms contamination but also terrestrial gas and liquid contamination. In addition of its operation in stringent cleanliness, it should also operate under the strict guidelines of the UN space treaties on Planetary Protection. It should also definitely allows the sample to be studied following requirements from the 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 from the container/sample catcher [residual gas sampling should be considered here], and then (5) to transfer (non-hazardous) 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, "life detection" tests, to determine whether the samples are hazardous, will have to be conducted.

A conceptual design of facility should include several distinct, but in a way connected (i.e., via pass boxes and/or doors) 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.),
(4) an opening laboratory (equipped with (Viton gloves) steel cabinet(s) under conditions of pure, positive pressure nitrogen),
(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 (or a combination of both?); this important point to be considered 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 inconvenient that will have to be further investigated.

For some samples, in particular for those from 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 sensible samples, and all the appropriate changes to the design and operation of the facility will be done.

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). Knowing that one of the main requirement and 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 minimize contamination.
Air-handling system is a major point 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 a best. All should also be done to minimize vibrations which can be problematic when manipulating small 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. Ultraviolet neutralization lamps and/or alpha-ray neutralizer of ^{210}Po radioactive source, to compensate electrostatic charging, are also a must.
From a contamination point of view, only a very restricted variety of materials can be introduced inside the cleanrooms and in particular within the (Viton gloves) cabinets, typically only stainless steel (304 and 316), pure aluminium and specific aluminium alloy, quartz glass and PTFE [+ Viton in case of Viton gloves cabinets] 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. Materials that possibly off-gas into the cleanrooms and especially within the (Viton gloves) cabinets should be avoided and/or carefully controlled.

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

The facility should be equipped with a system allowing communication, audio and video, in between persons from inside and also outside the different laboratories and facility; An access to the internal server of the facility should also be available, to be able to update in real time the sample database.

Outside of the cleanrooms (in the basement of the building?), equipment such as pumps for the vacuum systems, ultra pure water supply system, nitrogen purifiers, etc. should be incorporated in the general design of the facility.

More generally, office space for the permanent staff, as well as for guest staff, should be incorporated in the general concept. Whenever possible transparent apertures should be incorporated in the design of the cleanrooms/laboratories to allow a maximum of the visiting scientists (and general public) to discover the facility without having to physically enter critical parts of the laboratories.

The entire facility should be designed in a way to ensure a maximum 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.

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 (i.e., several years before planned return sample date) and that such a facility will have to preserve (and protect) samples for decades of research to be carried out on them.

In addition, the need of a suitable and secured remote (facility / "dead mode") storage of a representative subset (% TBD) of each of the curated extraterrestrial samples (to be stored in sealed steel cans within nitrogen-flooded steel cabinets) 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.

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 storages 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 (*gas that can be typically derived from the evaporation of ultra-pure liquid nitrogen*) [importantly, the amount of especially water vapour, oxygen and noble gases contained in the nitrogen gas should be monitored].
The temperature and pressure conditions should in fact already be kept "as low as possible" (i.e., P and T should in fact be as close as possible to the conditions that applied prior to their collection) already during the atmospheric entry and during all following steps, and should be monitored (with specific loggers) [*It is known that even a brief increases in temperature can result in conspicuous modifications of the characteristics of the samples*]. Currently, almost all curated extraterrestrial

samples are stored at room temperature (only a very small fraction of samples are preserved at "sub-freezing temperatures"). Curating frozen samples introduces significant challenges, but the appropriate storage temperature (and environment) will have to be first defined and then maintained in time. Routine curatorial procedures, especially sawing (to a lesser extend 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 (see WP6).

The (possible) contamination should also be monitored with specific "contamination witness materials/coupons" to be already placed inside the sample catcher/container on the spacecraft, and to be renewed and controlled until arrival inside the curation laboratory.

Another point to be considered is the magnetic conditions/properties of the samples; So far this is not really taken into account; Knowing that it can already be affected directly by the spacecraft propulsion system(s), and then during the atmospheric re-entry, landing, transportation to the facility and then also within the facility. Appropriate measures will have to be defined. For future missions, ways to protect the sample from the spacecraft propulsion system should be encouraged. In every case, the disruption of magnetic properties should be quantified with the use of analogue materials and large magnetic fields sources should be (as much as possible) banned from the facility.

In term of monitoring, possible "weathering"/alteration of the samples during their stay inside the facility should also be controlled. A need of 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 samples integrity within the facility, it cannot be totally excluded that some alteration (of whatever type) of the sample will occur.

The environment of the (Viton gloves) steel cabinets, as well as the surface of tools and materials to be in (direct) contact with the samples will have to be carefully monitored and controlled (the use of specific mass spectrometers should be envisaged for this purpose?).

Different types of sample holders and storage containers are possible/should be envisaged, depending on the specificities of the samples and subsamples, such as in 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 envisaged and designed based on the specificities of the samples to be curated and on the specific requirements from the scientific community and from the WP4]. When applicable, the sample return containers can be used for storage. For long term storage all the sealed containers should be secured in locked nitrogen (Viton gloves) 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.

For the allocation of samples, specific transport sample containers/holders will have to be designed due to the differences in the requirements of the different types of analyses to be conducted (in link with WP6).

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/allocation of a limited amount of samples 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 start already 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 insure proper monitoring of the contamination (already during the construction of the sampling device(s)).

Importantly, each of the (to be) collected and (to be) curated samples have a unique (and distinct) history and come from (somewhat) 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.

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 (Viton gloves) 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.

For the recovery of the samples from the sample catcher/container ("recovery container") and transfer to the "curation container(s)", different methods have been used in the past, depending on the size of the sample, including picking-up directly the samples from the catcher/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 (electrostatically controlled) micromanipulators. On top of the manipulations themselves, proper approaches of characterisation and storage of such small samples will have to be developed.

At first an initial processing and characterization (/documentation) of the samples should be conducted, including naming (a sample ID is given to each sample), 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 closed/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 [FIB]), X-ray fluorescence, microRaman spectroscopy, etc. should be envisaged (knowing that the extent of the preliminary examination (to be completed within the facility) 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 (pre-)selection of appropriate samples for research, before submission of a loan request. This would not only allow to reduced handling and limit unnecessary manipulations, but it would also prevent unnecessary loans.

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 bandsaw operated without blade lubrication (i.e., dry cutting; knowing that in this 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. New 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, (thin) sections, FIB foils, etc. will have to be prepared; 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.

<u>Short list of the identified main requirements and important issues to be especially investigated</u>

*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 at 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 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.

Additional requirements for the curation of samples from Mars

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 biosafety level 4 containment (BSL4; the highest level of 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.

BSL4 requirements

BSL4 facilities are designed to contain the most hazardous microbiological agents on Earth and to prevent them 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.

Conflicts between BSL4 and curation facilities

As already discussed in previous paragraphs of this report, curation of extraterrestrial samples requires an extremely clean environments (with particularly low levels of chemical contaminants and no biological or organic contaminants) involving the use of positive pressure isolators. BSL4 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.

Possible solutions

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.

References

Abe, M., Yada, T., Uesugi, M., Karouji, Y., Nakato, A., Kumagai, K., and Okada, T. 2015. Current status of JAXA's Extraterrestrial Sample Curation Center (abstract #1245). 46th Lunar and Planetary Science Conference. CD-ROM.

Allen, C., Allton, J., Lofgren, G., Righter, K., and Zolensky, M. 2011. Curating NASA's extraterrestrial samples—Past, present, and future. Chemie der Erde 71:1–20.

Allen, C., Allton, J., Lofgren, G., Righter, K., Zeigler, R., and Zolensky, M. 2013. Curating NASA's extraterrestrial samples. Eos 94(29):253–254 [16/07/2013].

Allwood, A., Beaty, D., Bass, D., Conley, C., Kminek, G., Race, M., Vance, S., and Westall F. 2013. Life detection in extraterrestrial samples. Astrobiology 13(2):203–216.

Beaty, D. W., Allen, C. C., Bass, D. S., Buxbaum, K. L., Campbell, J. K., Lindstrom, D. J., Miller, S. L., Papanastassiou, D. A. 2009. Planning considerations for a Mars Sample Receiving Facility: summary and interpretation of three design studies. Astrobiology 9(8):745–758.

Bell, M. S. and Allen, C. C. 2005. Cleanroom robotics – Appropriate technology for a sample receiving facility? (abstract #1395). 36th Lunar and Planetary Science Conference. CD-ROM.

Berthoud, L., Schroeven-Deceuninck, H., Vrublevskis, J., Guest, M., Baker, R., Bridges, J., Crook, B., Grady, M., Pope, A., Sephton, M., Sims, M., and Smith, C. 2013. Concept for a lunar and asteroid sample return facility. Proceedings of the 64th International Astronautical Congress, Beijing, China. IAC-13-D3.2.4.

Bridges, J. C. and Guest, M. 2011. Planetary protection and Mars sample return. Proceedings of the Institution of Mechanical Engineers, Part G: Journal of Aerospace Engineering 225(2):239–246.

Fletcher, L. A., Allen, C. C., and Bastien, R. 2008. Curation of frozen samples (abstract #2202). 39th Lunar and Planetary Science Conference. CD-ROM.

Hilts, R. W., Skelhorne, A. W., and Herd, C. D. K. 2012. Creation of a cryogenic, inert atmosphere sample curation facility: establishing baselines for sample return missions (abstract #5352). 75th Annual Meeting of the Meteoritical Society.

Holstein, J. L., Pelker, E. A., and Heck, P. R. 2011. Pilot digitalization project for the meteorite collection at the Robert A. Pritzker center for meteoritics and polar studies (abstract #5323). 74th Annual Meeting of the Meteoritical Society.

Kearsley, A. T., Smith, C. L., Spratt, J., Benedix, G. K., Hunt, A., Russell, S. S., Joy, K. H., and Gounelle, M. 2011. Meteorite polished sections: X-Ray map imagery for documentation, curation and virtual-loan' of irreplaceable materials (abstract #5280). 74th Annual Meeting of the Meteoritical Society.

Neal, C. R. 2000. Issues involved in a Martian sample return: integrity preservation and the Curation and Analysis Planning Team for Extraterrestrial Materials (CAPTEM) position. Journal of Geophysical Research 105(E9):22,487–22,506.

Race, M. S., Allton, J. H., Allen, C. C., and Richmond, J. Y. 1999. Planning considerations for Mars sample return containment. Journal of the American Biological Safety Association 4(2):53–59.

Sandford, S., Bajt, S., Clemett, S., Cody, G., Cooper, G., Degregorio, B., DeVera, V., Dworkin, J., Elsila, J., Flynn, G., Glavin, D., Lanzirotti, A., Limero, T., Martin, M., Snead, C., Spencer, M., Stephan, T., Westphal, A., Wirick, S., Zare, R., and Zolensky, M., 2010. Assessment and control of organic and other contaminants associated with the Stardust sample return from Comet81P/Wild2. Meteoritics and Planetary Science 45:406–433.

Velbel, M. A. 2014. Terrestrial weathering of ordinary chondrites in nature and continuing during laboratory storage and processing: Review and implications for Hayabusa sample integrity. Meteoritics and Planetary Science 49(2):154–171, doi: 10.1111/j.1945-5100.2012.01405.x.

Yada, T., Fujimura, A., Abe, M., Nakamura, T., Noguchi, T., Okazaki, R., Okada, T., Ishibashi, Y., Shirai, K., Zolensky, M. E., Sandford, S., Uesugi, M., Karouji, Y., Ueno, M., Mukai, T., Yoshikawa, M., and Kawaguchi, J. 2011. Hayabusa sample curation at planetary material sample curation facility in JAXA (abstract #5386). 74th Annual Meeting of the Meteoritical Society.

Yada, T., Fujimura, A., Abe, M., Nakamura, T., Noguchi, T., Okazaki, R., Nagao, K., Ishibashi, Y., Shirai, K., Zolensky, M. E., Sandford, S., Okada, T., Uesugi, M., Karouji, Y., Ogawa, M., Yakame, S., Ueno, M., Mukai, T., Yoshikawa, M., and Kawaguchi, J. 2014. Hayabusa-returned sample curation in the Planetary Material Sample Curation Facility of JAXA. Meteoritics and Planetary Science 49(2):135–153, doi: 10.1111/maps.12027.

Zolensky, M., Nakamura-Messenger, K., Fletcher, L., and See, T. 2008. Curation, spacecraft recovery, and preliminary examination for the Stardust mission: a perspective from the curatorial facility. Meteoritics and Planetary Science 43(1/2):5–21.

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Biosafety in Microbiological and Biomedical Laboratories (BMBL). US Department of health and Human Services, Public Health Service, Centers for Disease Control and Prevention and national Institutes of Health, 5th Edition. Washington, DC: US Government Printing Office, 2009.
[http://www.cdc.gov/biosafety/publications/bmbl5/]

Biological agents: the principles, design and operation of Containment Level 4 facilities. Advisory Committee on Dangerous Pathogens. Published by the Health and Safety Executive.
[http://www.hse.gov.uk/pubns/web09.pdf]

Laboratory biosafety manual. World Health Organization, Geneva. 3rd Edition. 2004, 186 pages, ISBN 92 4 154650 6.
[http://www.who.int/entity/csr/resources/publications/biosafety/Biosafety7.pdf?ua=1]