# Development of Capabilities and Instrumentation for Curation and Analysis of Returned Samples

A White-Paper for the NRC Planetary Science Decadal Survey, Reflecting the Viewpoints of the NASA Analysis Group CAPTEM (Curation and Analysis Planning Team for Extraterrestrial Materials)

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<sup>\*</sup>This white paper has been posted on the CAPTEM website noted above and inputs to it were sought from the planetary science community at large. Numerous comments and suggestions were received from many members of this community, and resulted in substantial revisions and endorsements to this white paper.

#### INTRODUCTION

Laboratory analysis of samples returned from Solar System bodies allows a wealth of information to be obtained about the processes involved in their formation and evolution. Not only can measurements be made with precision and sensitivity far beyond what can be done remotely, but a significant fraction of samples can be preserved for future generations to study as laboratory capabilities evolve. The case for pursuing sample return missions to the Moon, Mars, comet nuclei and asteroids is made in other white papers (from CAPTEM and others). Here we discuss the rationale for the support of the critical requirements of curation and instrument development for PI-led laboratories around the US associated with these sample return missions. In this context, it is important to note that, among planetary exploration missions, sample return missions are unique in that they require a well-developed infrastructure and capabilities for the appropriate curation and analyses of the returned materials to realize the science objectives of these missions.

# SUPPORT AND DEVELOPMENT OF CURATION CAPABILITIES FOR RETURNED SAMPLES

Curation is the critical interface between sample return missions and laboratory research. Its role is to maintain the integrity, provenance, ground truth and context for scientific questions and other planetary (orbital and surface science) data sets. Proper curation maintains and increases the scientific value of the samples over time. As a case in point, the Apollo lunar samples are still the subject of cutting-edge research forty years after collection because they have been carefully preserved, meticulously documented, and responsibly allocated. Each of the five distinct collections of extraterrestrial materials currently curated by NASA (Apollo, Antarctic meteorites, interplanetary dust particles (IDPs), Genesis solar wind samples, and Stardust cometary and interstellar dust particles) continues to be extensively utilized by researchers around the world (in FY08, over 1,300 samples were allocated from these collections) and new discoveries continue to be made based on analyses of these samples (see the following link for examples of the kinds of significant discoveries being made based on analyses of planetary materials: http://www.psrd.hawaii.edu/Archive/Contents.html).

In this section, we discuss the status of the five distinct collections of planetary materials currently supported by NASA, and the anticipated challenges for the appropriate curation of current and future returned samples.

#### **Status of Curation of Current Collections**

The Astromaterials Acquisition and Curation Office at the Johnson Space Center (JSC) is responsible for the curation of NASA's collections of extraterrestrial samples. Curation includes documentation, preservation, preparation, and distribution of samples for research, education, and public outreach. The currently curated collections include: (1) the Apollo lunar rocks and soils; (2) meteorites collected from Antarctica; (3) cosmic dust collected in the stratosphere; (3) solar wind collected by the Genesis mission; and (5) comet and possible interstellar particles collected by the Stardust mission. In addition, JSC will have the opportunity to curate a subset of any asteroid particles collected by the JAXA Hayabusa mission, scheduled to return to Earth in 2010.

#### Apollo Lunar Rocks and Soils

The samples returned by Apollo 11, 12, 14, 15 16 and 17 represent a total of 382 kg of rocks and soils (with over 100,000 subsamples). The curatorial facility for the Apollo samples

(i.e., JSC Lunar Sample Laboratory) comprises dedicated cleanroom laboratories with nitrogen gloveboxes, where samples are kept at low O<sub>2</sub> and H<sub>2</sub>O partial pressures (<10 ppm). It includes a high security storage vault and sample preparation laboratory. Precision cleaning techniques are used for all tools and equipment utilized in this curatorial facility. An extensive database has been developed to support sample selection and tracking, and there is a data library that includes detailed records for each sample. Special shipping and receiving procedures and protocols have been established for the Apollo samples. All Apollo lunar samples, regardless of condition, are returned to JSC following the completion of approved research. Returned samples are documented and stored in a dedicated laboratory, and these samples are available for allocation to other scientists. Finally, the curation plan for the Apollo samples also includes remote storage of 14% of the collection at a dedicated Remote Storage Facility, within the NASA White Sands Test Facility.

# Antarctic Meteorites (originating from asteroids, Moon, Mars)

On an annual basis, 200 to 1,500 meteorites are collected by the Antarctic Search for Meteorites field teams and shipped in a frozen state to JSC. Since 1976 over 17,000 meteorite samples have been recovered and returned to JSC. The curatorial facility for Antarctic meteorites at JSC, where these frozen samples are subjected to controlled warming to ambient temperature, comprises dedicated Class 1000 cleanroom laboratories with nitrogen gloveboxes and flow-benches, and includes the capabilities for sample preparation for various types of analyses. This facility provides for secure storage of ~8000 samples; long-term storage of the Antarctic meteorites is provided by the Smithsonian Institution's National Museum of Natural History. On an semiannual basis, published descriptions of all new samples collected during the previous few Antarctic field seasons are provided to the scientific community at large. An extensive database has been developed to support sample selection and tracking, and there is a data library that includes detailed records for each sample. Special shipping and receiving procedures and protocols have been established for the Antarctic meteorite samples. The collection receives on average >100 requests from different researchers in any given year, and allocates 300 to 600 individual samples a year.

# Cosmic Dust (originating from comets, asteroids)

The cosmic dust collection consists primarily of dust samples collected from the stratosphere by NASA aircraft; samples in this collection include those collected during dedicated flight campaigns to sample specific comets (meteor streams) and asteroid dust streams. The cosmic dust curatorial facility is a dedicated cleanroom laboratory that provides for secure sample storage and includes sample preparation capabilities. Published descriptions of all newly collected samples are provided to the scientific community at large. A database has been developed to support sample selection and tracking, and there is a data library that includes detailed records for each sample. Special shipping and receiving procedures and protocols have been established for cosmic dust samples.

# Genesis Solar Wind Samples

The Genesis solar wind samples include solar wind atoms implanted into high-purity materials (including silicon, silicon carbide, sapphire, diamond, germanium, gold, and aluminum) brought back by NASA's Genesis Discovery mission; the collection comprises over 10,000 collector fragments that resulted from spacecraft impact. The Genesis curatorial facility includes a dedicated Class 10 cleanroom laboratory that provides for secure sample storage and instrumen-

tation for sample characterization (ellipsometer, FTIR, automated microscope). The curatorial facility includes the capabilities for sample preparation and cleaning with ultrapure water that were developed specifically for the Genesis samples. An extensive photographic catalog (characterizing the collector condition and identifying the regime of the solar wind collected) has been prepared of these samples. A database has been developed to support sample selection and tracking, and there is a data library that includes detailed records for each sample. Special shipping and receiving procedures and protocols have been established for the Genesis samples.

#### Stardust Comet and Possible Interstellar Particles

NASA's Stardust mission returned cometary dust particles (from Comet Wild 2) (and possibly also interstellar dust particles) imbedded in aerogel and aluminum foil collector materials. The Stardust curatorial facility has a dedicated cleanroom laboratory that provides for secure sample storage. The curatorial facility includes specifically developed capabilities for cleanly extracting particles and particle tracks from the aerogel. An extensive catalog of these samples, which includes the results of all analyses that have been conducted thus far, is in preparation. A database has been developed to support sample selection and tracking, and there is a data library that includes detailed records for each sample. Special shipping and receiving procedures and protocols have been established for the Stardust samples.

# **Future Challenges for the Curation of Returned Samples**

In recent years, sample return mission proposals involving a wide variety of targets have been submitted under NASA's Discovery, Mars Scout, and New Frontiers Programs, and it is anticipated that the coming decade of Solar System exploration will include sample return from one or more targets of interest. The Announcements of Opportunity for these programs currently specify that the samples will be curated at JSC and that all curation costs, from inception of the mission until two years following Earth return, are to be borne by the mission. For most future sample return missions, dedicated curation laboratories will need to be designed and constructed, appropriate to the specific types of samples to be collected. In this context, it is important to note that for each of the five existing distinct collections, dedicated curatorial facilities, each involving substantial development of sample handling and preparation capabilities, were required for the appropriate curation of these collections. In particular, there may be specialized requirements in the future, for example, long-term preservation of ices and other volatiles, which would require storage and manipulation of such samples at sub-freezing temperatures.

Robotic and human exploration of the Moon is expected to involve the return of samples for research, infrastructure development, and resource utilization. These materials may be curated and distributed in an expanded version of the JSC Lunar Sample Laboratory. Specialized requirements may include compositional screening of large numbers of rocks and preservation of volatiles.

A future Mars Sample Return mission will impose the dual requirements of sample integrity and planetary protection. Mars samples are expected to be screened for evidence of possible life and biohazards in a dedicated sample receiving facility (SRF) with the combined characteristics of biosafety and curation laboratories. If the samples can be shown to contain neither life nor hazard, or if portions can be effectively sterilized, they may be released from the SRF for curation and distribution. Specialized requirements may include robotic sample handling, preservation of temperature-sensitive minerals, and preservation of atmospheric samples.

Finally, it is noted that the curation personnel at JSC, in the decades since the Apollo missions, have developed key experience and expertise in appropriate curation practices and sample processing and preparation skills for a variety of planetary materials. While there is certainly a need for the support of curation facilities for current and future returned samples, it is also essential that appropriately trained personnel remain involved in these curation as well as sample processing and preparation activities. Therefore, adequate support is essential for (1) the continued hiring and training of such personnel in the coming decade (commensurate with the requirements of the current collections of planetary materials and any additional materials anticipated to be brought back by future sample return missions during this time) and (2) development and advancement of sample processing and preparation techniques appropriate for the different types of Solar System materials that are likely to be returned in the next decade.

# INSTRUMENTATION DEVELOPMENT FOR ANALYSIS OF RETURNED SAMPLES

In this section we discuss the rationale for the development of advanced instrumentation for study of current and future returned samples.

# **Instrumentation Development in the Apollo Era**

The Apollo Program and the prospect of return of samples from the Moon drove a surge in laboratory instrumentation technology in the late 1960s. These developments were funded from a variety of sources, including government and private sources. Major improvements were made in a number of areas. The precision of isotopic analyses, principally with thermal ionization mass spectrometers (e.g., Wasserburg et al., 1969; Albee et al., 1970), significantly improved, driven by interest in high precision chronology of small samples of lunar rocks. Noble gas mass spectrometry was also advanced in the 1960s and the <sup>40</sup>Ar-<sup>39</sup>Ar dating method was developed (e.g., Turner, 1970). Neutron activation analysis was the primary tool for trace element analysis at the time and high resolution Ge-Li detectors were developed in the late 1960s, enabling development of instrumental neutron activation analysis methods, in which many trace elements could be measured without having to do chemical separations on samples (e.g., Goles et al., 1970). The electron microprobe, also developed in the 1960s, was, and continues to be an invaluable tool for studying mineral chemistry at the micrometer scale (e.g., Keil et al., 1970).

# **Instrumentation Development for Stardust and Genesis**

The Genesis and Stardust missions, the only completed sample return missions of the modern era, have similarly driven the development of advanced laboratory instrumentation, often of new types unimaginable in the Apollo era. Each of these missions drove analytical technology in different ways. NASA recognized the importance of advancing analytical cosmochemistry and initiated the Sample Return and Laboratory Instrumentation and Data Analysis Program (SLRI-DAP) several years before the return of the first mission. SRLIDAP funded both PI-built advanced instruments, such as the MegaSIMS and SARISA (see below) and the acquisition of commercial instruments that were significantly more capable than the then-current state-of-the-art and entailed significant development efforts by companies such as Cameca and FEI.

#### Genesis

The Genesis mission exposed high purity materials to solar wind for 26 months and returned (rather abruptly) to Earth in September 2004. Solar wind has relatively low energy, so it was implanted in these materials to shallow depths, only a few tens of nanometers. Two flagship

instruments were developed to do high precision depth profiling of the Genesis targets, and were funded through the Genesis mission and SLRIDAP.

The MegaSIMS, developed at UCLA, is a hybrid of a secondary ion mass spectrometer and an accelerator mass spectrometer (Mao et al., 2008). It was developed with the specific goal of measuring the oxygen isotopic composition of solar wind implanted in collectors flown on the Genesis mission. The development was driven by the need to eliminate the interference of <sup>16</sup>OH on <sup>17</sup>O in solar wind implants that have huge H/O ratios. The instrument succeeded and preliminary results were presented at the Lunar and Planetary Science Conference in March, 2008. The results are extraordinary: the Sun appears to be 5% richer in <sup>16</sup>O (McKeegan et al., 2009) than most samples known, including the Earth, Moon, Mars, most Comet Wild 2 dust, all bulk meteorites and most objects within those meteorites, the sole exception being some minerals in refractory inclusions in meteorites and, of all places, in Wild 2 dust.

The low concentrations of many elements place a premium on sensitivity of depth profiling measurements. The SARISA instrument, built at Argonne National Laboratory, uses lasers to resonantly ionize sputtered neutral atoms from Genesis targets (Veryovkin et al., 2004, 2008). These photoions are then extracted and mass analyzed with a time-of-flight mass spectrometer. This technique allows almost complete freedom from isobaric interferences, since only the element of interest is ionized, and very high sensitivity, since sputtering yields mostly neutral atoms, not ions. The SARISA instrument has a measured useful yield (atoms counted in the detector per atom removed from the surface) of ~25%, far better than state-of-the-art secondary ion mass spectrometers.

The Genesis mission has also benefited from NASA investments in commercially built instruments, including secondary ion mass spectrometers, noble gas mass spectrometers, and inductively coupled plasma (ICP) mass spectrometers. The Genesis mission also stimulated significant investment by European countries in laboratories at the Centre de Recherches Pétrographiques et Géochimiques-Centre National de la Recherche Scientifique, Nancy, France; ETH Zürich, Switzerland; and at the Open University, Milton Keynes, United Kingdom.

#### Stardust

The Stardust mission returned dust from Comet 81P/Wild 2 and perhaps contemporary interstellar dust in January, 2006. In anticipation of the successful return of these samples, NASA made a major effort to upgrade American laboratories through SRLIDAP (now the Laboratory Analysis of Returned Samples (LARS) Program); SRLIDAP also funded the development of novel sample preparation techniques to meet the challenges posed by the Stardust aerogel collectors (Westphal et al., 2004). A number of new instruments were acquired and played (and continue to play) major roles in studying Wild 2 dust. Several secondary ion mass spectrometers significantly advanced the state-of-the-art: two Cameca NanoSIMS 50L instruments at the NASA Johnson Space Center and the Carnegie Institution of Washington were the first built with larger radius magnets and are capable of detecting more masses simultaneously; a Cameca ims-1280 at the University of Hawaii is the first instrument in the US to have an imaging detector for high precision isotopic mapping. Advanced transmission electron microscopes were installed at the NASA Johnson Space Center, the University of Washington and Lawrence Livermore National Laboratory that offer unprecedented resolution in imaging and chemical mapping. Upgrades to synchrotron X-ray facilities at Brookhaven National Laboratory and Argonne National Laboratory have been partially supported by NASA. A new instrument that operates by the same principles as the SARISA instrument, but incorporates many improvements, is now under construction at the University of Chicago. It will permit chemical and isotopic analysis at lateral resolutions of a few nanometers with a useful yield approaching 50% (Davis et al., 2009).

The Stardust mission has provided some remarkable and surprising results. Comet 81P/Wild 2 contains abundant crystalline material formed at high temperature (Brownlee et al., 2006; Westphal et al., 2009), including a CAI (Simon et al., 2008) and objects that closely resemble chondrules (Nakamura et al., 2008). The oxygen isotopic compositions of these objects are similar to those in meteorites, implying that radial transport in the solar nebula was more effective than previously recognized (e.g., Ciesla, 2007).

# **Future Sample Return Missions**

One sample return mission is now flying. Hayabusa touched down on asteroid Itowaka and may have collected material from its surface. Although Hayabusa is a Japanese mission, NASA is participating in the mission and has been promised a share of any returned samples. In addition to Hayabusa, a number of missions to asteroids, comet nuclei, and the Moon are now under consideration by NASA through the New Frontier line of competitive, PI-led missions and, perhaps somewhat farther in the future, a sample return mission to Mars. Although the investment in instrumentation for Genesis and Stardust will be very useful for analyzing samples returned from these objects, the requirements for each of these kinds of objects are quite different from those of the two recent successful sample return missions. For example, the chronology of samples from the Moon and Mars will be studied by high precision mass spectrometry. Significant advances have been made in the last decade in these instruments, both for thermal ionization- and plasma ionization-type instruments, and improvements in these facilities will be needed to make the most of samples from differentiated objects. Lunar samples and perhaps asteroids can provide a rich record of exposure to solar wind and cosmic rays, and instruments to study this record, such as noble gas mass spectrometers and accelerator mass spectrometers will be needed. Samples from Mars, comet nuclei and perhaps asteroids may be of great importance for understanding low-temperature modification of high temperature materials. Study of volatiles would be key to comet nucleus sample return.

Sample return from a variety of solar system bodies is feasible in the next decade. The direction and rate of laboratory instrument development will be driven by the sequence and timing of sample return missions, but there is no question that past NASA support of instrument development has yielded great benefits in understanding the solar system and that further investment in laboratory facilities is key to furthering solar system science.

#### CONCLUSION/RECOMMENDATION

We must invest in sample handling and curatorial capabilities for future sample return missions and we must continue to advance the state-of-the-art in laboratory instrumentation to make the most of those returned samples. This requires funding of appropriate R&A programs and of state-of-the-art instrumentation and facilities. Such a program is currently supported by the Discovery Program Office. It is important for this support to continue and to be expanded to the New Frontiers Program missions.

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