

Hayabusa Sample Investigator's Guidebook: For the International Announcement of Opportunity

April 2, 2018

Hayabusa samples for the International Announcement of Opportunity

The Astromaterials Science Research Group (ASRG) of JAXA will handle the International Announcement of Opportunity (AO) with peer-review process in which Hayabusa sample investigation proposals will be selected. These guidelines are intended to help researchers who plan to submit proposals in response to the AO.

The key milestones for the International Hayabusa AO are as follows.

Announcement of Opportunity and start of receiving proposals: April 2, 2018

*There is no due date for proposals submission.

Note: All samples (or a Sample Information Sheet for consumed samples) allocated in the previous International AO must be returned before PIs will receive a new sample. A successful proposal may be turned down if the PI of the proposal has not returned samples until the new samples could be distributed.

Each time we would receive a proposal, it will be reviewed by at least two members of the ASRG who hold PhDs for assessment about the scientific merit of each proposal. The reviewers will judge whether it should be accepted for sample distribution or not, and the review results will be presented to the meeting which all the staff of the ASRG should join and discussed for decision for its sample distribution. As the ASRG would reach the conclusion for distribution, the curator will send an e-mail about the result of reviews to the PI. Thus, it will take around a month since the ASRG would receive the proposal that the ASRG will send the results of reviews to the PI. When the ASRG cannot reach the conclusion about the distribution, the ASRG will ask its steering committee, which consist of established planetary science researchers inside and outside JAXA, about the decision for the distribution. In this case, it will take more than a month since the ASRG would receive the proposal to send the e-mail of reviews' results to its PI. The unique attributes of the Hayabusa samples, for example, non-exposure to the Earth's air, pristine memory of the surface environment of the asteroid, the first regolith sample from an

asteroid, and so on will be preferred for the sample distribution. The proposer's demonstrated capability of handling and analyzing tiny particles and access to the required analytical facilities, the availability of the requested samples, and the feasibility of the proposed investigation will also be assessed. Forming consortia to maximize scientific output is especially encouraged for studies that will result in damage or destruction of the samples.

A maximum of five samples can be allocated to a proposed investigation. Based on the requirements of the proposed investigation, a proposer should select samples adequate for their research and indicate in the proposal the sample IDs of the selected samples. While the maximum number of samples to be allocated is five, a proposer can select up to 10 sample IDs to be indicated in the proposal. Listing additional samples and indicating the priority of each sample ID are very useful if an alternate sample needs to be allocated. And you should note that the sample catalog would be updated around the end of month even after the announcement. In these updates, newly described particles will be added to the list and distributed particles will be deleted from it.

The ASRG and its steering committee will decide which samples are to be allocated based on the proposal evaluation and the priority indicated by the proposers.

Record of the 1st International AO

For the 1st International AO, the AO committee received 31 proposals and selected 17 proposals for sample allocation. Sixty-two samples were distributed to the investigators. Among these, 36 were newly retrieved particles while 26 were particles returned from Preliminary Examinations (PEs). The successful proposals are as follows.

| PI | Title |
|-----------------------------------|---|
| Evelyn Füri | Noble gas (He-Ne-Ar) and nitrogen study of asteroidal dust grains returned by the Hayabusa mission |
| Hugues Leroux | Transmission electron microscopy study of (1) crystal defects in olivine and high-Ca pyroxene (2) Fe-Mg ordering in low-Ca pyroxene |
| Noriko Kita, Daisuke Nakashima | High-precision oxygen isotope analyses of Itokawa silicate mineral |
| Lindsay P. Keller | Coordinated analysis of Hayabusa mission samples |
| Takaaki Noguchi | An in-depth analysis of space weathering on Itokawa |
| Akira Tsuchiyama | Surface and 3D structures of Itokawa regolith particles for understanding surface processes on Itokawa |
| Thomas J. Zega | Microstructure and chemistry of Hayabusa soils |
| Tomoki Nakamura | Temperature, pressure and timing of a catastrophic impact event experienced by the Itokawa parent asteroid |

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| Kunihiko Nishiizumi | Measurements of cosmic-ray produced nuclides in Hayabusa samples |
| Fred Jordan | Argon isotopic compositions of Itokawa grains: Insights into the impact history and exposure ages of a rubble-pile LL chondrite asteroid |
| Wataru Fujiya | Irradiation history and solar wind composition recorded in the Hayabusa samples inferred from light element and noble gas analyses |
| Cécile Engrand, Lydie Bonal | Multidisciplinary investigations of Itokawa samples returned by the Hayabusa mission |
| Keisuke Nagao | Noble gas study combined with TEM and reflectance spectroscopic observations for further understanding of regolith processes on Itokawa |
| Henner Busemann | The characterization of the asteroid Itokawa regolith - A correlated study by X-ray tomography, micro-Raman spectroscopy and high-sensitivity noble gas analysis |
| Falko Langenhorst | Shock metamorphism and space weathering of Hayabusa regolith – Clues to the collisional history of an asteroid surface |
| Takashi Mikouchi | Mineralogy and crystallography of Hayabusa particles by synchrotron radiation X-ray and electron beam analyses: Reconstruction of Itokawa parent asteroid(s) |
| Monica M. Grady | Relationship between comets, asteroids, and meteorites |

Record of the 2nd International AO

For the 2nd International AO, the AO committee received 18 proposals and selected 16 proposals for sample allocation. Fifty samples were distributed to the investigators. Among these, 43 were newly retrieved particles while 7 were particles returned from PEs. The successful proposals are as follows.

| PI | Title |
|---------------------|--|
| Wataru Fujiya | Extended study on the irradiation history of the Itokawa asteroid by light element and noble gas analyses |
| Henner Busemann | Unraveling the history of asteroid Itokawa's regolith - by Raman and FTIR spectroscopy X-ray tomography and noble gas analysis |
| Kunihiko Nishiizumi | Studies of Itokawa's surface exposure by measurements of cosmic-ray produced nuclides |
| Kentaro Terada | Chronological studies on Itokawa particles |
| Jisun Park | ⁴⁰ Ar/ ³⁹ Ar age-dating studies of the regolith of asteroid 25143 Itokawa |
| Keisuke Nagao | Solar cosmogenic and trapped noble gas study of individual Hayabusa grains for further understanding of regolith processes on Itokawa |
| Tomoki Nakamura | The nature of a catastrophic impact event experienced by the Itokawa parent asteroid |
| Takaaki Noguchi | Unveil the irradiation history of each Itokawa particle: A combined study of transmission electron microscopy and noble gas mass spectrometry |
| Takaaki Noguchi | High-resolution three-dimensional structure of blisters on an Itokawa sample: Scanning transmission electron microscopy characterization using computed tomography |
| Akira Tsuchiyama | Studies of surface processes on Itokawa by systematic examination of surface features of Itokawa regolith particles |
| Arnold Gucsik | Clarification of shock history of asteroid Itokawa using cathodoluminescence and micro-Raman spectroscopy |
| Hisayoshi Yurimoto | Space weathering analysis of Itokawa particles by in-situ three dimensional noble gas distribution using novel sputtered neutral mass spectrometry |
| Ryan Ogliore | O & ²⁶ Al- ²⁶ Mg isotope systematics space-weathering effects and detailed characterization of Hayabusa samples |
| Edward Cloutis | Single grain reflectance spectroscopy of Hayabusa mineral grains |
| Mutsumi Komatsu | Detailed examination on metamorphic grades of Itokawa particles: Relation to |

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| | their original asteroid(s?) |
| Fabrice Cipriani | Investigation of regolith grains properties under simulated space environments |

Record of the 3rd international AO

For the 3rd International AO, the AO committee received 16 proposals and selected 12 proposals for sample allocation. Forty-nine samples were distributed to the investigators. Among these, 38 were newly retrieved particles while 11 were particles returned from PEs or previous AOs. The successful proposals are as follows.

| PI | Title |
|------------------------|---|
| Rhonda M. Stroud | Aberration-Corrected STEM quantitative analysis of nano-scale composition variations in space-weathered Hayabusa particles |
| Akira Miyake | A STEM-EELS study of carbonaceous material embedded in an Itokawa particle |
| Michael Zolensky | Measurements of shock effects recorded by Hayabusa samples |
| Lydie Bonal | Asteroidal space weathering: New insights from the investigation of the optical properties and microstructure of single Hayabusa grains |
| Queenie Hoi Shan, Chan | An investigation of the organic content and distribution of biomolecules in Itokawa particles |
| Takaaki Noguchi | Have the Itokawa particles with macrosteps on the surfaces experienced severe heating during their history? : A combined study of electron microscopy and noble gas mass spectrometry |
| Takaaki Noguchi | Asteroid Itokawa and LL chondrites: Oxygen isotopes and X-ray absorption mapping |
| Hikaru Yabuta | Search for carbon from the asteroid Itokawa particles |
| Lindsay P. Keller | Coordinated analysis of Hayabusa mission samples |
| Fred Jourdan | Further insight into the collisional history of asteroid Itokawa: Integrating petrography, EBSD and $^{40}\text{Ar}/^{39}\text{Ar}$ dating of dust particles |
| Kentaro Terada | Chronological studies on Itokawa particles 2 |
| Suporn Boonsue | Investigation and characterization of Itokawa regolith: Particle interaction processes |

Record of the 4th international AO

For the 4th International AO, the AO committee received 10 proposals and selected 8 proposals for sample allocation. Thirty-six samples were distributed to the investigators. Among these, 19 were newly retrieved particles while 17 were particles returned from PEs or previous AOs. The successful proposals are as follows.

| PI | Title |
|-------------------|---|
| Hiroshi Naraoka | Organic characterization of Hayabusa category 3 particles using desorption electrospray ionization/Orbitrap mass spectrometry (DESI/Orbitrap-MS) |
| Takaaki Noguchi | How long was the dwell time of each Itokawa grain near the surface of the asteroid Itokawa?: A combined study of high voltage electron microscopy and noble gas mass spectrometry |
| Maitrayee Bose | Volatile content and composition of the Itokawa surface |
| Falko Langenhorst | Analytical transmission electron microscopic study of pyroxene grains from the regolith of asteroid 25143 Itokawa: mineralogy, space weathering and shock |

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| | metamorphism |
| Rosario Brunetto | FTIR micro-tomography of Hayabusa grains |
| Josep Trigo-Rodriguez | Mechanical properties, magnetism and microporosity of Itokawa asteroidal regolith |
| Jisun Park | Continued $^{40}\text{Ar}/^{39}\text{Ar}$ age dating of the regolith of Asteroid 25143 Itokawa |
| Kuljeet K. Marhas | Al-Mg isotope systematic in pristine particles from Itokawa |

Record of the 5th international AO

For the 5th International AO, the AO committee received 3 proposals and selected 3 proposals for sample allocation. Nine pristine samples were distributed to the investigators. The approved proposals are as follows.

| PI | Title |
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| Luke Daly | Nanometer-Atomic scale observation of space weathering phenomena on Itokawa grains |
| Ming-Chang Liu | Analysis of light isotopes of implanted solar wind in Itokawa pristine particles: implications for solar isotopic compositions |
| Devin L. Schrader | Constraining formation and alteration conditions of asteroid Itokawa via microstructure and elemental compositions of sulfides from Hayabusa particles |

General notes on Hayabusa samples

A summary of the sample retrieval activity is given here. It includes information on samples that are not available for the International AO but which may be available in future opportunities.

The characteristics of particles available for the International AO are just as same as those for the International AO. The majority of the particles range in size from 20 to 50 micrometers. Category 1 and 2 particles have been identified as silicate type. Category 3 particles are identified as those including carbon. These categorizations are performed via Scanning Electron Microscope equipped with Energy Dispersive X-ray Spectrometer (SEM-EDS) analysis by the Curator. Category 1 and 2 particles are thus all dominated by silicate minerals and are the typical particles found in the sample catcher.

The sample catcher is composed of two rooms, Room A and Room B. For Room A, three types of retrieval method were employed: Method (1), scratching by a Teflon spatula; Method (2), direct pick-up by a probe; Method (3), free-fall onto a quartz disk. For Room B, methods (2) and (3) were employed.

Of the 3,300 particles recovered from Room A, 1,500 were confirmed by method (1) to be silicate type. These very tiny particles (mostly smaller than 10 micrometers) are attached to a Teflon spatula, and we have not yet tried to remove them.

More than 100 particles from Room A and the top cover of Room B were collected by

method (2). Method (2) was tried for Room A before method (3) was tried. The top cover of Room B was separated from the sample container before the particles inside the container were dropped onto a quartz disk by method (3). Method (2) is much easier to apply to the top cover than to the container itself because of the flat geometry of the cover compared to the complicated three-dimensional structure of the container. The particles, however, are much less visible than particles on a quartz disk because of the cover's rough surface.

We had not known how many more particles remain attached to the top cover of Room B. After the 2nd International AO, we succeeded to directly observe the surface of the top cover of Room B using SEM-EDS. Until now we observed almost all the area of the top cover of Room B and confirm the presence of more than 3,000 particles that are larger than 15 micrometers. Among them, a percentage of Itokawa particles is around 20%. The result of direct SEM-EDS observation of the top cover is reported in the following paper.

References of SEM-EDS observation

Yada et al. (2014) MetSoc abstract #5239

It is estimated that about 1,000 particles from Room A and 500 particles from Room B were deposited onto quartz disks. Approximately half of these are considered contaminant particles (mostly Al debris from the catcher surface). Room A particles range in size from 10 to 300 micrometers; Room B particles range from 10 to 200 micrometers. Of these particles, more than 700 were collected and subjected to SEM-EDS analysis (including a small number of particles that were lost afterwards). Of these, some particles were submitted to the PEs.

It is not presently clear how many more particles remain unrecovered inside the sample catcher.

Initial descriptions based on the SEM-EDS analysis permits sorting of the recovered samples into four categories.

Category 1: Particles with only ferromagnesian silicate composition features.

Category 2: Particles with ferromagnesian silicate plus other mineral features, such as metals, sulfides, and oxides.

Category 3: Particles with mainly carbon signatures.

Category 4: Particles likely to be contaminants, namely, debris from the parts within the sample catcher, showing composition features such as Al, quartz glass, and stainless steel.

Category 1, 2 and 3 particles obtained by both method (2) and (3) from Room A and Room B, and they have been allocated in the International AO.

The nature of Category 3 particles is puzzling and investigated by preliminary examination team and consortium team led by the curator. The results are shown in the references and the summary sheet of each sample in the catalog. The evidence that these samples are of extraterrestrial origin has not been discovered until now.

References of Category 3 particles Analysis

Ito et al. (2014) EPS 66:91

Kitajima et al. (2015) EPS 67:20

Naraoka et al. (2015) EPS 67:67

Uesugi et al. (2014) EPS 66:102

Yabuta et al. (2014) EPS 66:156

Some particles will be available as processed particles returned from previous studies. In the PEs, these particles have been subjected to X-ray CT scans, noble gas analysis, XRD/XRF analysis, SEM/TEM/EPMA analysis, SIMS analysis, a search for organic material, and so on. The results of previous studies will be described in data sheets that will be made public upon release of the AO. One data sheet is attached to each of the particles returned from previous studies. Proposals for further detailed analysis on top of the already obtained results are expected. The past analysis history of each particle is presented in the data sheets, since some analyses may constrain the scope of meaningful future analysis to be performed on some particles.

References of PEs

Ebihara et al. (2011) Science 333, 1119-1121.

Nakamura et al. (2011) Science 333, 1113-1116.

Nagao et al. (2011) Science 333, 1128-1131.

Naraoka et al. (2012) Geochemical Journal, 46, 61-72

Noguchi et al. (2011) Science 333, 1121-1125.

Tsuchiyama et al. (2011) Science 333, 1125-1128.

Yurimoto et al. (2011) Science 333, 1116-1119.

Nakamura et al. (2012) PNAS, 10.1073/pnas.1116236109

Ebihara et al. (2015) MAPS 50, 243-254.
Nakamura et al. (2014) MAPS 49, 215-227.
Noguchi et al. (2014) MAPS 49, 188-214.
Noguchi et al. (2014) MAPS 49, 1305-1314.
Tanaka et al. (2014) MAPS 49, 237-244
Tsuchiyama et al. (2014) MAPS 49, 172-187.
Wakita et al. (2014) MAPS 49, 228-236.

Other particles will be available as pristine particles. Only rather general properties known from SEM and BSE imaging results and EDS spectrum information from a few points on each particle will be available in the Sample Catalog for these particles.

Guidelines for Proposers

PIs could not receive samples for the International AO, if they could not return samples distributed to the same PIs in the previous international AO(s). An otherwise successful proposal may be rejected if this condition is not met.

The Hayabusa samples are valuable and irreplaceable samples returned from an asteroid. Since they are tiny particles that require careful and dedicated treatment, proposers must first demonstrate their capability of handling and successfully analyzing such tiny particles. The proposers must be ready to receive samples at the time of they actually propose, which is scheduled to be the end date of sample distribution by the Curator.

The time-consuming nature of the sample retrieval process limits the total number of particles available for the AO. The total number of samples available to investigators is not very large, so each investigator is limited to a maximum of five samples. Moreover, JAXA curation team has a sample preservation policy that one half of each of five size-bins (<50 μ m, 50-100 μ m, 100-150 μ m, 150-200 μ m, and 200 μ m<) of pristine particles from each RA and RB will be preserved and not available for the international AO. Consortia of scientists proposing multiple analyses for individual samples are highly encouraged. In addition to describing the scientific goals and analysis plans for the samples, sample request proposals must also detail the size and number of the samples, along with specific features of the samples that are being requested. Sample requests must also detail all requirements regarding the sample transfer method.

Each particle has its own ID number indicated in the Sample Catalog so that a proposer

can explicitly specify the particles requested. Proposers must also state what properties of the requested particles match their proposed investigations. Each proposer should select up to 10 sample IDs as candidates that match the requirements of the proposed investigation, in case more than one investigator requests the same particle. If all of the samples indicated in the proposal have been assigned to other proposals that have higher priorities, however, the proposal will be rejected.

Here are some notes on the Sample Catalog. The available Sample Catalog for the International AO consists of three lists. The first is the list of pristine particles in Category 1 and 2. The second one is the list of processed particles in Category 1 and 2. The third one is the list of particles in Category 3. The main table lists the basic properties of the samples including sample ID, sample size, sample category (Category 1-4, see above), mineral phase, and, for the processed particles returned from PEs, a list of the analyses to which the particle has been subjected. When a sample that matches a proposer's interest is found, the proposer should follow the links for further information (SEM images and EDS spectrum for pristine particles, and a detailed data sheet for a processed particle returned from PEs) to complete the proposed research plan. Please refer to the SEM images and EDS spectra carefully; do not refer only to the phase list in the Catalog.

Since the Curator is prepared to package pristine samples for transfer to an investigator's institution in a high-purity nitrogen (ppb levels of trace gases) atmosphere, samples can reach the investigator without exposure to the earth's air if necessary. However, such precautions may not be necessary for some proposed investigations. Thus sample request proposals must describe the exact environmental conditions for the sample transfer procedures to be made by the Curator. On the other hand, processed particles will be transferred to the investigator in the final condition described in the sample catalog.

A proposer who wants to have allocated samples sent with a special container must explain in the proposal why the samples should be transported with the special container. The PI must send the special container to JAXA as soon as it would receive an e-mail of selection for sample distributions from JAXA. JAXA that is responsible for minimizing the degradation of Hayabusa samples will decide the sample transfer environment based on the investigator's request and the cleanliness level of the special container. If the researcher could not send the container one month after it would have received the selection e-mail, the JAXA standard container will be used for its distribution.

An allocated sample must be returned to the Curator as soon as the proposed investigation

is completed. If a proposed investigation will completely consume samples, this fact must be clearly stated in the proposal. An investigator cannot transfer samples to other investigators/institutions unless this action was described in the original approved sample request, or without explicit approval of the Hayabusa Curator.

Delivery to NASA

The delivery of Hayabusa samples to NASA opens a research opportunity provided by NASA in parallel to JAXA's International AO.

Record:

Fifteen particles were allocated to NASA in December 2011.

Ten particles were allocated to NASA in January 2013.

Five particles were allocated to NASA in March 2016.

Five particles were allocated to NASA in June 2017.

Investigator's Responsibility

Hayabusa Sample Investigators are responsible for the security of the samples allocated to them and will be held personally accountable in the event of sample loss. Investigators must make every effort to avoid unnecessary contamination of the samples and must return allocated samples to the Curator immediately upon completion of the approved investigation. The Curator assumes one year as the standard sample allocation period but this can be modified according to the actual progress of a particular investigation, at the discretion of the Curator. The investigator who hopes to have extended period for their study must resubmit an investigation plan sheet to the Curator and receive an agreement from the Curator. Extension can be up to twice and each extension period is shorter than a year. When a proposed investigation is of consortium type that involves multiple institutes, the proposer can propose a two-year investigation plan. For such a study, a single extension for less than a year is allowed.

Investigators must report the results of their analyses to the Curator in a timely manner. This is particularly critical for samples that have not received significant characterization by the Curator.

Investigators must notify the Curator as they would divide the allocated samples into multiple pieces or slices. Then the Curator will give sub-numbers for each of pieces and/or slices and inform them of their numbers. The investigators must use those given

sub-numbers for the pieces and slices of the samples.

Investigators are required to report the results of all sample analyses to the Curator very soon after completion of their investigations, even if these results are not ultimately published. Investigators must send a summary sheet reporting their investigation one year after the receipt of samples even if their study is not completed. A Sample Summary Sheet containing the minimum information that an Investigator should report to the Curator will be posted upon release of the AO. Investigators have complete control over publication, but they are requested to notify the Curator in a timely fashion of any publication that results from their analysis of the Hayabusa samples. When it is judged useful for a future allocation, unpublished results may be added to the sample analysis report submitted to the Curator. The Curator is interested in constructing a knowledge warehouse that stores all information related to Hayabusa Sample Investigation to aid future sample investigations. The Sample Catalog, which a proposer should carefully inspect before drafting a proposal, will be enhanced by input from the Investigators and will better serve future research opportunities by allowing more sophisticated study plans to be proposed. In their publications, proposers can include Acknowledgements in the following format.

The Hayabusa-returned samples XXXX (sample ID) were allocated by JAXA's ASRG as part of its distribution for the international announcement of opportunity for research.

JAXA holds a Symposium of Solar System Materials every year since 2013. Investigators must report their result or current status of their research at this symposium.

Hayabusa Sample Investigators are responsible for arranging for the transfer of samples from ISAS to their institutions. While the Curator will prepare JAXA-standard containers for the transfer, investigators can also consult the Curator if they prefer to use their own containers. Various methods of transfer may be considered, such as hand carrying and FedEx. It is noted, however, that some sample transport routes have the risk of unintentional opening by various persons. While JAXA is willing to lend some assistance, investigators are responsible for going through all customs formalities. Investigators are requested to cover all transfer fees.

All of the terms and conditions of the Hayabusa Sample AO are described below.