Environmental Assessment in the Pre-launch Phase of Hayabusa2 for Safety Declaration of Returned Samples From Asteroid (162173) Ryugu: Background Monitoring of Possible Contaminants During Development of the Sampler System

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Abstract

We report the ground-based environmental assessments during development of the sampler system until the launch of the Hayabusa2 spacecraft. We conducted static monitoring of potential contaminants to assess the environmental cleanliness during (1) laboratory work throughout the development and manufacturing of the sampler devices, (2) installation of the sampler system on the spacecraft, and (3) transportation to the launch site at the JAXA's Tanegashima Space Center. Major elements and ions detected in our inorganic analysis were sodium (Na), potassium (K), and ionized chloride (Cl^-); those were positively correlated with the total organic content and with exposure duration in the range from 10^1 to 10^3 nanogram per monitor coupon within a ~ 30 mm-diameter scale. We confirmed that deposits on the coupon were totally less than the microgram-scale order during manufacturing, installation, and transportation in the pre-launch phase. The present assessment yields a nominal safety declaration for sample analysis of the pristine sample (>5 g) returned from asteroid (162173) Ryugu combined with a highly clean environmental background level. We expect that the Hayabusa2-returned sample from Ryugu without severe and/or unknown contamination will allow us to provide native profiles recorded in the carbonaceous asteroid history.

Introduction

The re-entry capsule of Hayabusa2 spacecraft was successfully retrieved from Woomera, South Australia, on December 6, 2020. Hayabusa2 made comprehensive surveys of C-type near-Earth asteroid (162173) Ryugu (e.g., Watanabe et al., 2019; Sugita et al., 2019; Kitazato et al., 2019; Grott et al., 2019; Jaumann et al., 2019; Arakawa et al., 2020; Morota et al., 2020; Okada et al., 2020; Kitazato et al., 2021; Tatsumi et al., 2021a, 2021b; Sakatani et al., 2021) with two touch-down operations for sampling. The Hayabusa2 sampler system worked as expected in two landing operations (e.g., Tachibana et al., 2014, 2021; Sawada et al., 2017; Okazaki et al., 2017—Appendix). The sample container, which contained the Ryugu sample, was sealed using the metal-seal system developed to maximize the scientific value of the sample, especially with regard to retention of volatile gas molecules from samples and prevention of terrestrial contamination (e.g., Sawada et al., 2017; Okazaki et al., 2017). If there is no severe and/or unknown contamination on samples, detailed sample analysis will provide us crucial information with the primordial native conditions of organics, water, and mineral interactions regarding key issues in the molecular evolution on the asteroid.

To assess the entire sampling process aimed at safeguarding the scientific value from significant and unknown contamination, the Hayabusa2 sampler team conducted (i) off-nominal operational assessments (e.g., projector operation: Takano et al., 2020; small carry-on impactor: Ito et al., 2021), and (ii) a laboratory-based environmental assessment aimed at monitoring the background cleanliness of each stage of the development of the Hayabusa2 spacecraft (e.g., see the assessment policy by Sawada et al., 2017). Focusing on the latter issues, this technical report describes the assessment results during the pre-launch phase (Table 1). Based on both quantitative and qualitative assessments, we can evaluate potential contaminants, which could have been artificially induced, seamlessly during implementation,

engineering development, launch, or capsule re-entry into the Earth's atmosphere. We can thus provide a robust final safety declaration for analysis of samples from pristine C-type asteroid Ryugu.

Overview of the Hayabusa2 sampler status for quality assurance

We designed a seamless monitor procedure for an efficient work flow up to the time of initial sample processes (Tachibana et al., 2014; Okazaki et al., 2017; Sawada et al., 2017). To formulate a safety-declaration protocol for the Ryugu sample (i.e., Tachibana et al., 2021; Yada et al., 2021a,b), Table 1 summarizes an overview of sample processes through the initial implementation, the engineering development, and the assessment on the axis of Hayabusa2 sampler team. To date, we have contamination knowledges from learned lessons from assessment of contaminants in the previous Hayabusa mission (e.g., category-3 particles; Uesugi et al., 2019; and references therein), potential contaminants from the Hayabusa2 off-nominal operations (e.g., Takano et al., 2020; Ito et al., 2021), the curation procedure and the cleanliness level at the JAXA Extraterrestrial Sample Curation Center (e.g., Sugahara et al., 2018; Yoshitake et al., 2021), and sample transportation container for Hayabusa2 samples (e.g., Ito et al., 2020; Shirai et al., 2020; Uesugi et al., 2020). We also highlight that there are literature sources for quality control and assurance for other sample-return projects (e.g., Dworkin et al., 2017; McCubbin et al., 2019; Chan et al., 2020; and references therein). As a practical assessment following Sawada et al. (2017), we deployed the contamination monitor coupon set nearby the sampler flight model for assessing how much Earth-derived terrestrial material potentially exist till the launch of Hayabusa2 spacecraft (Figure 1-a).

Environmental assessment based on static monitoring

Figure 1b–c represents the schematic design and appearance of the monitor coupon, containing a Pyrex container, an aluminum container, and carbon adhesive tapes for inorganic and organic assessments, and microscopic observation, respectively (see Sawada et al., 2017 for more details). The timeline and assessment plans for quality control, monitoring, collection, and storage during the development of the Hayabusa2 sampler are summarized in Figure 1d. To evaluate both the inorganic and organic background levels of the ambient environment, we deployed the monitor coupon during the assembly of the sampler system (Figures 2 and 3). For comparison with the static cleanliness test above, we also performed air-filter sampling (Multi-stage Andersen Cascade Impactor, Tokyo Dylec Corp.) and swab sampling using reference sources involving a quartz filter (8 cm diameter × 4 mm thickness) and glass wool (~0.3 g), respectively. The precipitate artifact during the thermal vacuum test for the spacecraft (Sawada et al., 2017) was also analyzed for the identification the volatile compounds (Supplementary information).

The analytical methods used for inorganic and organic assessments have been reported previously with regard to quantitative and qualitative evaluations (e.g., Takano et al., 2020; Ito et al., 2021). All glassware and aluminum materials were washed using organic solvent and flushed with ultrapure water, following Karouji et al. (2014). All glassware and aluminum parts used in the assessment were cleaned by heating (450°C for 5 hr) in air to remove any artifacts of organic contaminants (e.g., Takano et al., 2020).

Results And Summary

Inorganic and organic assessments

The concentrations of total organics, major elements, and major ions monitored by the coupons at different locations, occasions, and/or durations (coupon set code; CPS-1 to CPS-12) are summarized in Figure 4. Concentrations of elements, ions, and total organics were less than or on the order of 10^3 nano-gram (i.e., $1\text{ }\mu\text{g}$ or less) for all the coupons ($\sim 30\text{ mm}$ in diameter). Raw concentration of the elements (Fe, Cu, Ni, Cr, Zn, Na, K, Ca, Mn, Al, Ti, Mg, Co, Sn, Pb, V, and P), ions (including Cl^- , NO_2^- , Br^- , NO_3^- , SO_4^{2-} , PO_4^{3-} , and F^-), and organics are shown in Tables 2, 3, and 4, respectively. The concentrations normalized to surface area (i.e., ng cm^{-2}) can be obtained using the monitor coupon size (Figure 1-b).

The major inorganic elemental and ion species detected were sodium (Na), calcium (Ca), and potassium (K), chloride (Cl^-), sulfate (SO_4^{2-}), and nitrate (NO_3^-). Their profiles are positively correlated with total organic content in the range of 10^1 to 10^3 ng per monitor coupon. Figure 5 shows the time-dependent static accumulation of ambient aerial deposits onto the monitor coupons throughout the procedure (CPS-1–12, including STA1, SFA2, and VAB). There are clear positive correlations between total sodium, potassium, and chloride, and total organic contents, which closely approximate the 1:1 line. Both major inorganic ions and total organic fluxes converged in the range of 10^1 to 10^3 ng per monitor coupon, resulting in a static deposition amount of $<1\text{ }\mu\text{g}$ overall within $\sim 7.0\text{ cm}^2$ and $\sim 5.3\text{ cm}^2$ for aluminum schale and pyrex schale, respectively. Representative compositions of organic deposits are obtained by the gas chromatogram (Figure 6). A wide range of aliphatic hydrocarbons of straight-chain alkanes ($<n\text{-C}_{19}\text{H}_{40}$) were detected (e.g., extracted ion chromatogram, $m/z = 57$). The total amounts of other miscellaneous organics were quantified based on the corresponding response of the internal standard method (e.g., Takano et al., 2020). The exposure time-dependent accumulation profiles are similar to the results of the cleanliness test in the clean-room chamber at ISAS/JAXA (i.e., the nitrogen-circulation clean chamber; Sugahara et al., 2018).

Tables 5 and 6 summarize the basic statistics and the correlation matrix for the concentrations of major components in the static samples (i.e., CPS1–12 and CAS1–12; $n = 10$). Most profiles of the major components (e.g., Na, K, Cl^- , F^- , NO_3^- , Zn, SO_4^{2-} , Mg, and P) positively correlate with each other, except for the profile of aluminum (Figures 5e and S2). We note that we used clean-room-type aluminum foil, to store and carry the monitor coupons, which could potentially introduce a certain amount of aluminum onto the coupon. We also conducted microscopic observations of the solid contaminants to evaluate their morphological characteristics (Figure 7), and found that globular shape particles were more common (70%) than fibrous material (13%). Further non-destructive analysis using micro-Raman and infrared spectroscopy (e.g., Kitajima et al., 2015) at ISAS/JAXA can be performed on request.

We stored potential Earth-derived artifacts obtained from each procedure (Table 1) (i.e., the category-3 particles; Uesugi et al., 2019). Therefore, if necessary, we can provide a careful description, on request, as

suggested from the lessons learned from the previous Hayabusa mission with regard to high resolution small-scale mass spectrometry (e.g., NanoSIMS: Ito et al., 2014; ToF-SIMS: Naraoka et al., 2015) and spectroscopy (e.g., X-ray absorption: Yabuta et al., 2014). Interestingly, it should be noted that Chan et al. (2021) has reported a detailed description of carbonaceous and organic matters that are derived from Itokawa.

Safety declaration of the Ryugu sample processes

The Hayabusa2 mission succeeded in delivering its re-entry capsule to Earth in December 2020, and the total sample amount was confirmed to exceed 5 g (Tachibana et al., 2021; Yada et al., 2021a,b), including millimeter-sized grains to centimeter-sized pebbles. This study reports the pre-launch phase environmental assessment during the manufacturing of the sampler devices, their installation onto the spacecraft, and their transportation. We conclude that the exposure-time-dependent deposition profiles of potential contaminants were firmly underscored and similar to those obtained from the clean-room facility assessment (Sugahara et al., 2018). All the potential contaminants have been stored at the JAXA Extraterrestrial Sample Curation Center in a nitrogen-purged storage, and are available for future analysis on request.

The total amount of potential contaminants in the sampler system is estimated to be less than 100 ng cm⁻², which is much smaller than the total amount of returned samples. Along with the fact that there was no off-nominal situation from the spacecraft launch to the capsule re-entry to Earth, we conclude that ambient background artifacts have been characterized and will not affect detailed sample analysis.

We expect that the lessons learned in the process from implementation to practical knowledge during the last eight years will also represent a valuable baseline for future sample-return missions (e.g., Dworkin et al., 2017; Usui et al., 2020; Anand et al., 2020; Chan et al., 2020; Neveu et al., 2020; Farley et al., 2020; Fujiya et al., 2021).

Abbreviations

C-type: Carbonaceous-type; JAXA: Japan Aerospace Exploration Agency; ISAS/JAXA: Institute of Space and Astronautical Sciences/JAXA; ESCuC: The Extraterrestrial Sample Curation Center; TNSC: Tanegashima Space Center; TSC: Tsukuba Space Center; STA: Spacecraft Test and Assembly Building; VAB: Vehicle Assembly Building; SFA: Spacecraft and Fairing Assembly building; NASA: National Aeronautics and Space Administration; TDS: Thermal desorption system; GC: Gas chromatography; GC/MS: GC coupled with mass spectrometry; EIC: Extracted ion chromatogram; FT-IR, Fourier Transform Infrared Spectroscopy; NanoSIMS: Nano-scale Secondary Ion Mass Spectrometry; GAEA: GAs Extraction and Analysis system.

Declarations

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Authors' contributions

SK and YT led this work, conducted the assessment, deployed the coupon set, monitored the environment, interpreted the data, and finalized the manuscript. TN and RO supported the assessment process as witness scientists up to the launch of the spacecraft. HS and ST led the Hayabusa2 sampling system with the feedback from KS, YT, YM and RO. HS and SN performed the spacecraft's thermal vacuum test and collected the precipitate sample. HY, TY, MU and MA supervised the assessment scheme based on feedback from the previous Hayabusa mission. Hayabusa2 project team members confirmed the present assessment as regards the quality control and assurance for future sample processes. YT, HS, and ST designed the implementation of fundamental scheme. All authors contributed to the manuscript and approved the final version of the data report.

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Availability of data and materials

Please see the supplementary information.

Ethics approval and consent to participate

Not applicable.

Consent for publication

Not applicable.

Competing interests

The authors declare that they have no competing interests regarding the data reported in this document.

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Tables

Due to technical limitations, table 1 to 6 is only available as a download in the Supplemental Files section.

Figures

a

Figure 1

(a) Photographs of the Hayabusa2 spacecraft in the clean room at ISAS/JAXA (after Tachibana et al., 2014). (b, c) Schematic design and appearance of the monitor coupon equipped with an aluminum scale, Pyrex scale, aluminum disk, sapphire glass disk, aluminum square plate (#1, #2), and carbon adhesive tape (#1, #2) for the Hayabusa2 witness coupon (after Sawada et al., 2017). (d) Environmental assessment procedures for timelines, locations, and operational profiles from static monitoring. Blue and

green bars represent the installation time of the coupon and the nitrogen gas purge time into the sampler system, respectively. Abbreviations for locations are defined at the end of main text.

Figure 2

Representative snapshots of the environmental assessment. (a) Practical deployment of the monitor coupon in the processes of ISAS (ISAS-C building clean room) and TSC (JAXA Tsukuba Space Center), (b) STA1 building clean room at TNSC (JAXA Tanegashima Space Center), (c) SFA2 building clean room, (d) VAB building clean room at TNSC, and (e, f) the final launch site at TNSC.

Figure 3

Environmental monitoring near the Hayabusa2 spacecraft. (a) Sampler horn. (b) Sample container with witness coupon inside (Sawada et al., 2017). (c, d) Inside of the sample catcher before elimination of surface dust.

Figure 4

Concentrations of total organics, major elements, and major ions monitored by the monitor coupon (Nos 1–12, including STA1, SFA2, and VAB). The sample codes are listed in Tables 2 and 3. Each static, quantitative assessment has been normalized in units of nanograms per sample (ng / sample).

Figure 5

Representative profiles of the static accumulation of ambient aerial deposits onto the monitor coupons (Nos 1–12, including STA1, SFA2, and VAB): (a) Sodium (Na) and total organics, (b) potassium (K) and ionized chloride (Cl⁻), (c) zinc (Zn) and sulfate (SO₄²⁻), and (d) calcium (Ca) and ionized nitrate (NO₃⁻). (e) Principal component loading using the correlation matrix (Table 5) of the major components in the static monitoring data of CPS1–12 (n = 10) and CAS1–12 (n = 10), as shown in Table 6.

Figure 6

Representative extracted ion chromatograms (EICs) of the aliphatic hydrocarbon compounds (n-alkanes; $m/z = 57$ on EIC) obtained from online thermal desorption- gas chromatography/mass spectrometry (TD-GC/MS) with a quantitative internal standard method (Takano et al., 2020); (a) Typical terrestrial reference (humic acid as standard reference; from FUJIFILM Wako Pure Chemical Corporation), (b) STA1 (code: 20141024-QF-01-#1), (c) SFA2 (code: 20141024-QF-06-#1).

Figure 7

(a) Carbon adhesive tape used for the environmental assessment. (b–g) Representative microscopic observations of the morphologies of artifact materials on the coupon.

Supplementary Files

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