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Methods and tools for handling, transportation, weighing, and pelletization applied to the initial analysis of volatile components in the Hayabusa2 samples

Ryuji Okazaki[1*], Shinji Yamanouchi[2], Kazuhiko Shimada[3], Atsushi Baba[2], Fumio Kitajima[1] and Toru Yada[4]

Abstract

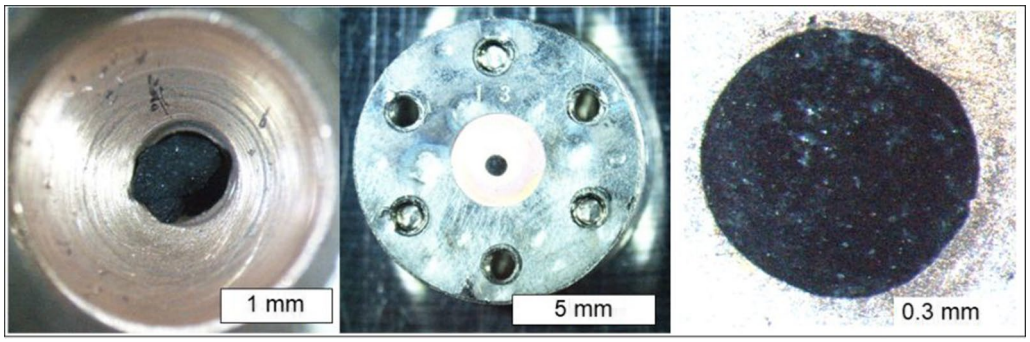
The Hayabusa2 spacecraft succeeded in sampling and returning materials from the C-type, near-Earth asteroid (162173) Ryugu. As part of the Hayabusa2 Initial Analyses, chemical and isotopic compositions of volatile species were measured. The samples analyzed were grains of about 1 mm in diameter and were individually treated without exposure to Earth's atmosphere throughout the entire analytical/experimental processes to minimize alteration and contamination effects by adsorption of Earth's atmosphere or chemical reactions with reactive species such as oxygen and water in Earth's atmosphere. In order to perform spectroscopic and electron-microscopic observations in advance of a series of the isotopic measurements, the sample surface needed to be smoothed. We employed a pelletization method to obtain the required flatness for the returned samples because pelletization is a less sample-consuming method compared to mechanical polishing, microtomy, or ion milling. In order to perform the subsequent analyses, the samples must undergo minimal contamination during the pelletization procedure and be easy to remove from the pelletization tools. Therefore, embedding with resins or low-melting-point metals was not employed. Under these constraints, tools and methods for sample pelletization, handling, and transportation were developed. The tools developed for pelletization and housing also contributed to easier handling of small (less than about 1 mm in diameter) samples. Here we describe the methods and the tools that enable treatment of pristine asteroidal samples under non-atmospheric exposure conditions throughout transportation, weighing, pelletization, and installation into the instruments for chemical and isotopic measurements. The methods and tools we developed can be applied to other small samples including meteorites, cosmic dust, and future returned samples.

Keywords: Hayabusa2, Pelletization, Weighing, Handling, Transportation, Volatile isotopes, Airtight

*Correspondence: okazaki.ryuji.703@m.kyushu-u.ac.jp

[1] Department of Earth and Planetary Sciences, Faculty of Sciences, Kyushu University, 744 Motooka, Nishi-Ku, Fukuoka 819-0395, Japan
Full list of author information is available at the end of the article

Graphical Abstract



Introduction

The Hayabusa2 spacecraft returned surface/sub-surface materials from the C-type, near-Earth asteroid (162173) Ryugu in 2020 (Tachibana et al. 2022). The returned samples are expected to have information that cannot be obtained from meteorite research because meteorites are inevitably altered during and after atmospheric entry (e.g., Bland et al, 2006; King et al. 2020). The elemental and isotopic compositions of volatile components in Ryugu samples are important to elucidate the origin and evolution of terrestrial volatiles, such as oceans, atmosphere, and life. In order to gain the maximum results from the return sample analyses, it is important that samples are unaltered as much as possible from the condition in which they were collected from the asteroid, including minimal contamination by terrestrial and artificial materials. One of the easiest ways is to apply simple and quick isotope analysis without any combination analysis, which minimizes the possibility and degrees of contamination. However, it is important to perform non-destructive analyses (e.g., mineralogical and spectroscopic studies) in advance of destructive analyses (chemical and isotopic measurements of trace elements), which allows us to study possible host phases and the evolutionary history of the volatile components. Electron/optical microscopic observations and spectroscopic observations are some of the basic and important analyses, but samples generally need polishing to obtain a flat surface for these analyses, which causes sample alteration by resin, abrasive compounds, and lubricant agent. Other methods for the preparation of polished sections, such as microtomy, ion milling, and electron polishing are limited by the sample size to which they can be applied, and can introduce contamination and losses of samples during the processing.

The Hayabusa2 mission led the initial analysis of Ryugu samples by six sub-teams (Tachibana et al. 2014). One of them, the volatile sub-team, carried out combined analyses of Fourier transform infrared spectroscopy (FTIR), field emission scanning electron microscope (FESEM) observations, secondary-ion mass spectrometry (SIMS), neutron activation analysis (NAA), and chemical and isotopic measurements of volatile components, such as inorganic compounds, nitrogen, and noble gases (Okazaki et al. 2021, 2022). Ryugu is thought to be a rubble pile asteroid that was created by the accumulation of fragments produced by impact to the original, larger parent asteroid, and hence Ryugu samples could be composed of the fragments with different lithologies. Therefore, a combination of non-destructive and destructive analyses enables us to obtain the maximum information about volatile components in samples with complicated lithologies, such as Ryugu samples.

Minimum sample size for isotopic measurements of Hayabusa2 volatiles

The minimum sample weight required for volatile analysis was calculated based on the concentration of primordial ^{132}Xe, which is commonly contained in chondritic meteorites and can be reasonably estimated to be $\sim 10^{-8}$ cm^3 g^{-1} at standard temperature and pressure (STP) (e.g., Mazor et al. 1970). The minimum amount of ^{132}Xe required for measurement with < 10% uncertainty for $^{128-136}$Xe/^{132}Xe isotopic ratios is $\sim 10^{-12}$ cm^3 STP ($\sim 3 \times 10^7$ atoms), corresponding to Ryugu samples of ~ 0.1 mg in weight. Stepped heating extraction needs a larger sample mass, expected to be ~ 10 times the minimum for 3–5 extraction steps, and hence the sample mass and size should be about 1 mg. Because the sample handling tools were designed and developed before the Hayabusa2's capsule returns to Earth, the desired sample mass of 1 mg was thought to be reasonable given that it corresponds to 1% of the nominal value of the total recovered mass of 100 mg (Tachibana et al. 2014)

(although the actual mass was 5.4 g; Yada et al. 2022). The sample size corresponding to 1 mg should be within a range of 0.9 and 1.3 mm in diameter for the 1.0 to 2.5 g/cm^3 densities that are possible for C-type asteroids and carbonaceous chondrites (Britt et al. 2002).

Tools and methods for volatile analyses

An overview of the analytical flow and the sample handling operations developed in this study is shown in Fig. 1. First, samples stored in two sapphire containers allocated by Japan Aerospace Exploration Agency (JAXA) were introduced into a glove box that was filled with pure nitrogen gas and installed at Kyushu University. Each of the samples was selected from the sapphire containers (Procedure 1), and located in the conical hole of a Cu disk of the pelletization tool (Procedure 2). Pelletized samples were stored in a container and transported to the Planetary Material Sample Curation Facility (PMSCF) of JAXA for FTIR and FESEM observations (Procedure 3). After these observations, the samples were removed from the pelletization Cu disks, measured for their masses (Procedure 4), and used for isotopic measurements (Procedure 5). Some of the pelletized samples were used for SIMS analysis after coating with osmium and platinum. After the SIMS analyses, the samples were removed from the Cu holders and mounted into the sapphire container [see "G) Sapphire container for samples handled in Earth's atmosphere"] in order to perform NAA and Ar–Ar dating (Procedures 6–8). Procedures 1–5 were performed under a pure-nitrogen environment (blue boxes in Fig. 1), whereas Procedures 6–8 were done in Earth's atmosphere (gray boxes in Fig. 1). The oxygen concentration in the used nitrogen gas (generated by a nitrogen generator, M4NT-0.4II, KOFLOC Corp.) was ~ 300 ppm, and the dew point in the glove box during the operation was about − 40 °C.

The most important requirement for the above sample handling operations and measurements was to minimize contamination of terrestrial and artificial materials. Therefore, the materials of the sample handling tools that came into direct contact with the samples (Table 1) were limited to those that were washable with organic solvents and are thermally resistant (i.e., can withstand 220 °C). In addition, the designs of the tools should be as simple as possible to reduce the risk of sample losses, which also reduces the possibility of the contamination from the tools during the operations. After repeated rehearsals, the designs of the tools were modified several times (3–10 times) to resolve potential issues due to static electricity or human errors during the operations.

In summary, the following points were considered in the development of the tools and procedures: 1) to

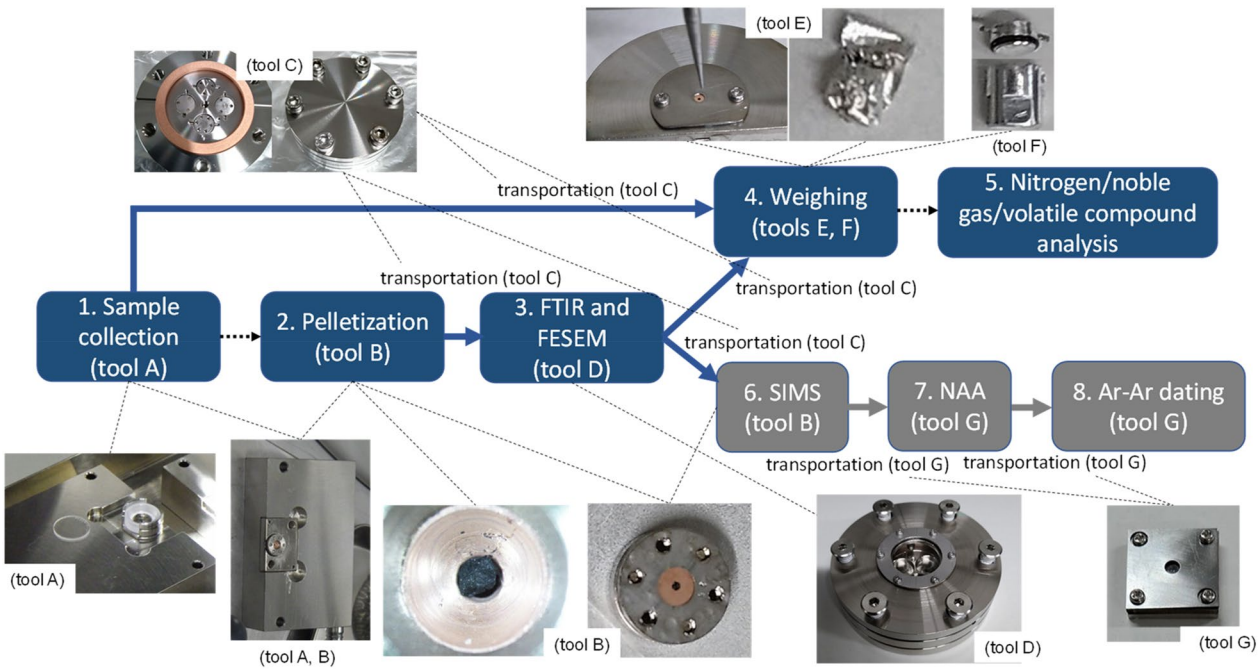


Fig. 1 Flow diagram of the volatile component analyses conducted by the Hayabusa2-Initial-Analysis Volatile sub-team. Operations 1–5 (blue boxes) were carried out without exposure to Earth's atmosphere, whereas operations 6–8 were done in Earth's atmosphere (gray boxes). The tools used in each operation are shown in the parentheses. The letters of the alphabet, tools "A–G", correspond to those of the titles of subsections in the main text (e.g., "tool A" refers to subsection "(A) Sample handling stage")

conform to the analytical conditions and instrumental constraints for the analyses, 2) to minimize contamination from the environment and materials used in the tools, 3) to reduce issued during sample handling operations, and 4) to be useable in a glove box filled with pure nitrogen gas.

The tools and instruments developed for use in each operation and measurement are as listed below: (A) Sample handling stage, (B) Pelletization tool and stainless-steel holder for pelletized samples, (C) Transportation container, (D) FTIR/FESEM adapter holder and CaF_2 viewport, (E) Sample encapsulation tool, (F) Weighing container, and (G) Sapphire container for samples handled in Earth's atmosphere.

(A) Sample handling stage

The handling stage was developed for safe and easy observation and collection of sample grains stored in the sapphire dish (dia. 15 mm) distributed by JAXA (Figs. 1 and 2, and Additional file 1). The stage was made of stainless steel (JIS: SUS304, ISO: 4301-304-00-I), and consisted of two assemblies: one to place the JAXA sapphire dish, and the other is used to place the pelletization tool. They can be connected together with the connecting plates (the top and middle columns of Fig. 2a). In order to encapsulate non-pelletized samples with a metal cup, the stage for the pelletization tool was replaced with another stage designed for the metal-cup holder of the encapsulation tool (the bottom column of Fig. 2a). The handling stage for the JAXA dish (the left assembly in Fig. 2a) was designed so that the basal plane of the JAXA dish had the same height as the surface plane of the diamond disk during the pelletization (the level shown by a red dotted line in Fig. 2b) or the same height as the top of the metal cup during the encapsulation (the level shown by a red dotted line in Fig. 2c).

(B) Pelletization tool

Before the destructive analyses of volatile components, we performed non-destructive FTIR and FESEM observations. In-situ measurements of light-element (hydrogen, carbon, nitrogen, oxygen, silicon, and sulfur) isotopes by SIMS were also carried out (Fig. 1). These analyses require a smooth sample surface, and the SIMS analysis offers the most stringent constraint on the smoothness of the sample surface, ideally being < ~0.3 μm (e.g., Thomen et al. 2010). In order to carry out the combined analyses as shown in Fig. 1, the pelletized samples should be transported to respective laboratories without damage. After the non-destructive analyses, the samples will be measured for their masses to obtain the concentrations of volatile components, such

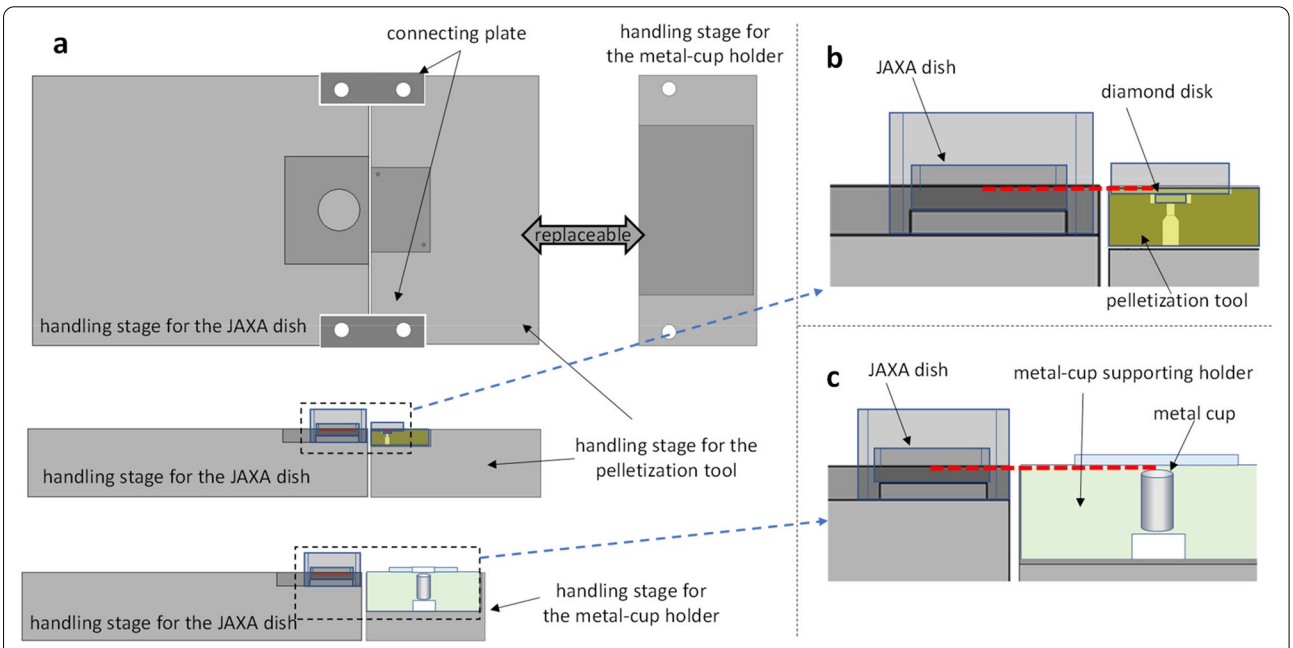


Fig. 2 Handling stage used for sample collection from the sapphire dishes distributed from JAXA. **a**. The left block was used to set the JAXA dishes, whereas the right block is replaceable for the pelletization or encapsulation. **b**. Enlarged view of the cross-section of the setting for the pelletization. A dotted red line shows the height of the samples in the JAXA dish and that of the diamond disk used for the pelletization. **c**. Enlarged view of the cross-section of the setting for the encapsulation. A dotted red line shows the height of the samples in the JAXA dish and that of the top of the metal cup

as noble gas and nitrogen concentrations. Therefore, the pelletized samples must be easily removable and retrievable from the preparation tool, and they must remain intact during the transportation. In order to comply with these requirements, we adopted a pelletizing technique, in which the sample was placed in a Cu disk and pressed against a diamond disk. The pressed sample was mounted on and stuck to the Cu disk and was resistant to breaking or fracturing. Our pelletization method is less sample-consuming and less contaminating than other techniques to prepare samples with a smooth surface, such as mechanical/electron polishing, microtomy, and ion milling; these methods require samples to be fixed with resin or tungsten deposition, which leads to contamination or considerable losses of samples (which may be up to about half of the sample).

The key to the pelletization operation is to place the sample in the proper position for pressing and ensure that the two planes pressing the sample are parallel. These adjustments to the positions and conditions of the sample and the tool assemblies are extremely complicated and difficult in a glove-box operation and are a source of failure and trouble. To address these issues, we designed the pelletization tool (Additional file 2) to place the sample in a double conical dimple of the Cu disk (pure copper) and to press the sample against the diamond disk (ϕ3 and t0.9; EDP Corp.) with the metal bead (made of stainless steel, JIS: SUS440C or ISO: 4023-440-04-I). With this design, the sample and the metal bead are naturally positioned at the center of the Cu disk, and hence the pressing pressure inevitably acts at the center (Fig. 3a). The metal beads were used by gradually replacing the larger ones with smaller ones, which enabled efficient pelletization. Also, two types of Cu disks were prepared for 0.5 mm and 1 mm-sized samples (Fig. 3b and c). The inner surface of the Cu disk was polished with diamond powder (9 μm in diameter) before use. All the assemblies were washed using an ultrasonic cleaner, in turns with neutral detergent, organic solvents (acetone and ethanol), and pure water, and were finally heated in a vacuum at 220 °C prior to the pelletization operation.

The flow of the pelletization operation is shown in Fig. 4. First, a stainless-steel holder with an outer diameter of 1 cm (named as 1 cm SS-holder; Fig. 5a) was fixed on the pelletization base (Fig. 4a). A diamond disk was placed at the center hole of the 1 cm SS-holder, and a Cu disk was mounted on the diamond disk. The diamond disk and the Cu disk were contacted together and fixed on the pelletization base using the disk-clamping plate (Fig. 4b). The assembled component of the pelletization was set on the sample handling stage (Fig. 2a and b), and a sample was placed into the Cu disk (Fig. 4b) from the JAXA dish. The metal bead was gently placed on the sample, and the top plate was fixed to the pelletization base (Fig. 4c). Finally, the sample was gradually pressed against the diamond disk in several steps using metal beads with different diameters. The metal beads used for the smaller Cu disk (Fig. 3b) were 1.0 and 0.8 mm in diameter, and those for the larger Cu disk (Fig. 3c) were of 2.5, 2.0, and 1.5 mm. The metal bead was pressed against the sample using the M4 pressing screw with a torque of 0.6 N (Fig. 4c). The pressed sample was tightly mounted on the

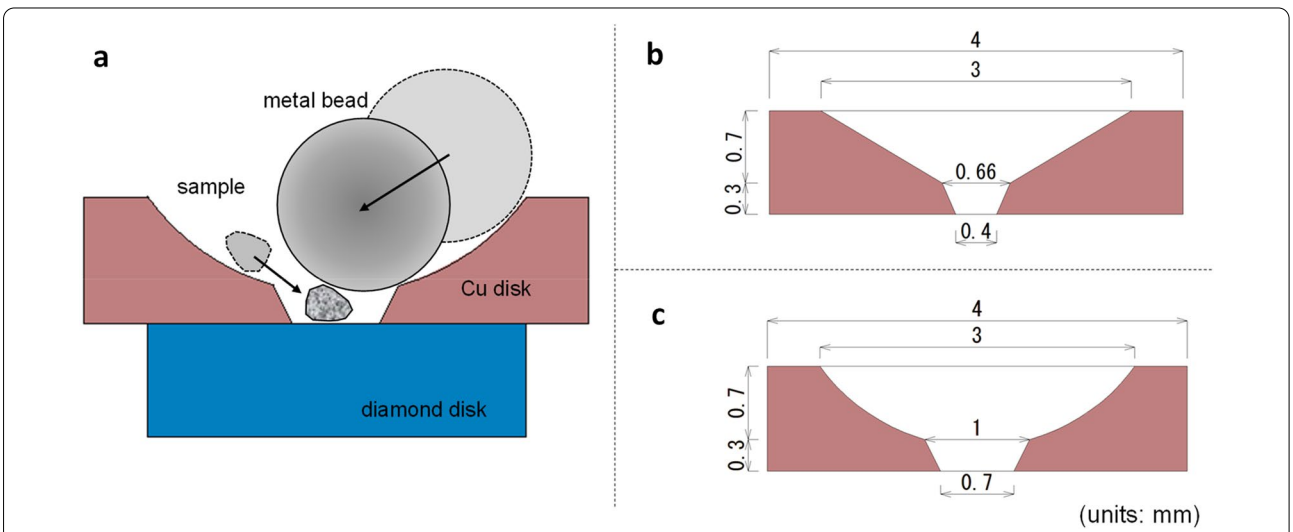


Fig. 3 Schematic diagram of the pelletization and cross-section images of the Cu pelletization disks for different sample sizes. **a**. A sample was placed in the conical hole of the Cu disk and pressed against the diamond disk with the metal bead. **b**. Cu disk for smaller samples of 0.5 mm diameter. **c**. Cu disk for larger samples of 1 mm diameter

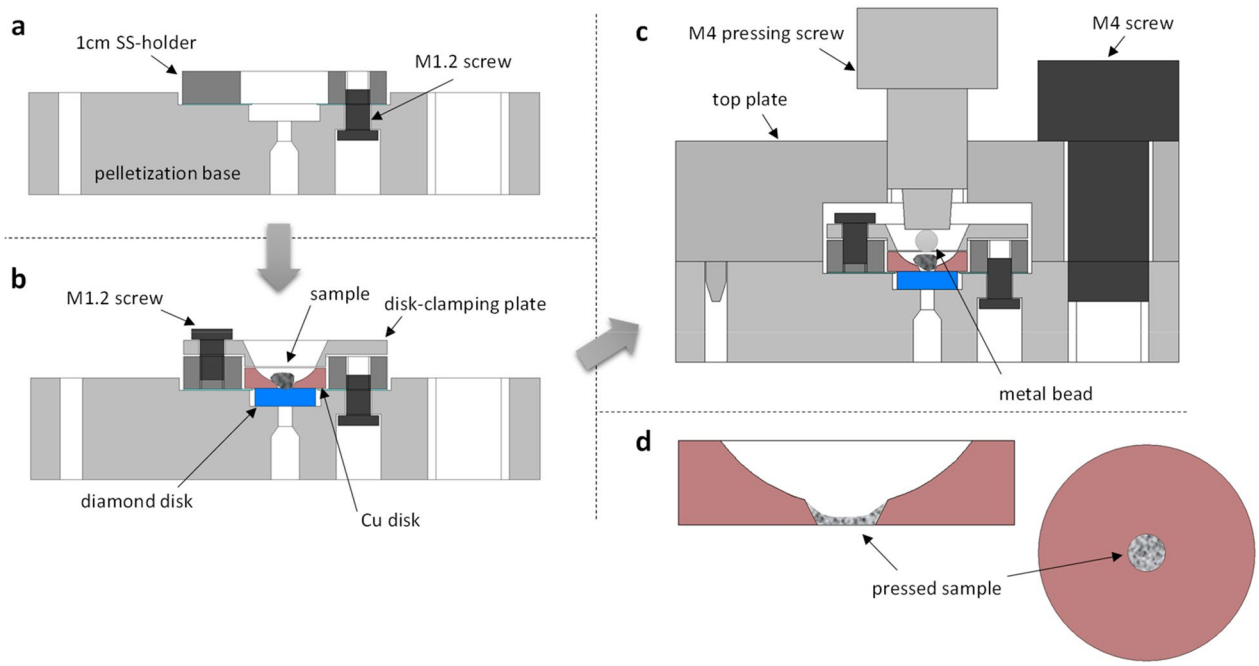


Fig. 4 Schematic diagram of the pelletization operation. **a**. A stainless-steel holder (shown as 1 cm SS-holder) was installed on the pelletization base and fixed using M1.2 (φ1.2) screws. **b**. The diamond and Cu disks were set on the pelletization base and fixed using the disk-clamping plate and M1.2 screws. A sample was placed against the Cu disk. **c**. The top plate was fixed using M4 screws. The sample was pressed against the diamond disk with a metal bead using the M4 pressing screw. **d**. Schematic diagram of the Cu disk holding the pelletized sample

Cu disk after the pelletization (Fig. 4d). The hole shapes/sizes of the Cu disks, bead sizes, and pressing force were adjusted to be appropriate for these sample sizes. Our pelletization method can be applied to different sample sizes using different setup conditions.

After the pelletization, the disk-clamping plate was removed and replaced with a spring-loaded lid (Fig. 5b) to fix the Cu disk in the 1 cm SS-holder (Fig. 5a). The height of the sample surface was set at a fixed position from the top of the 1 cm SS-holder (Fig. 5c) using a Mo face-plate of 50 μm thickness (Fig. 5a), which ensured the same surface height between different pelletized samples during FTIR, FESEM, and SIMS analyses. The 1 cm SS-holder closed with the spring-loaded lid was removed from the pelletization base. The holder can be easily handled during transportation, weighing, and the instrumental analyses.

Preliminary experiments were conducted to evaluate the pelletization tool. Powdered (smaller than ~ 30 μm in diameter) or submillimeter-sized grains of Murchison CM chondrite was used for the rehearsals. Samples of 0.1 and 0.3 mg were used for the small and large Cu disks (Fig. 3), respectively. The flatness of the pelletized sample surface was measured using the fine-focus knob of an optical microscope to be ± 5–10 μm at a center region of 200 by 200 μm^2 (Fig. 6). The outer edge of the pellet (i.e., the boundary between the pelletized sample and the Cu disk) was less smooth because of the pressing pressure lower than in the center of the pellet. Although the flatness of the pelletized samples on the scale of 200 μm in width was not ideal, on the scale of 25 μm it was good enough to determine the isotopic compositions of nitrogen or carbon hotspots with SIMS (Okazaki et al. 2021). A pelletization rehearsal was conducted in the N_2 glove box to confirm the feasibility of this operation. A transportation rehearsal using the pelletized Murchison samples was also conducted to confirm that the samples would not become broken or detached from the Cu disk during transportation by domestic and air mails using the transportation container described below.

(C) Transportation container

In order to transport samples between laboratories with minimal contamination, transportation containers must be airtight, easy to handle, and sturdy. Given the handling of samples in the glove box, the container should be of applicable size, and the screws used for the containers should be able to be installed and tightened with one hand.

To ensure sealing performance, the transportation container was made from a commercial vacuum flange,

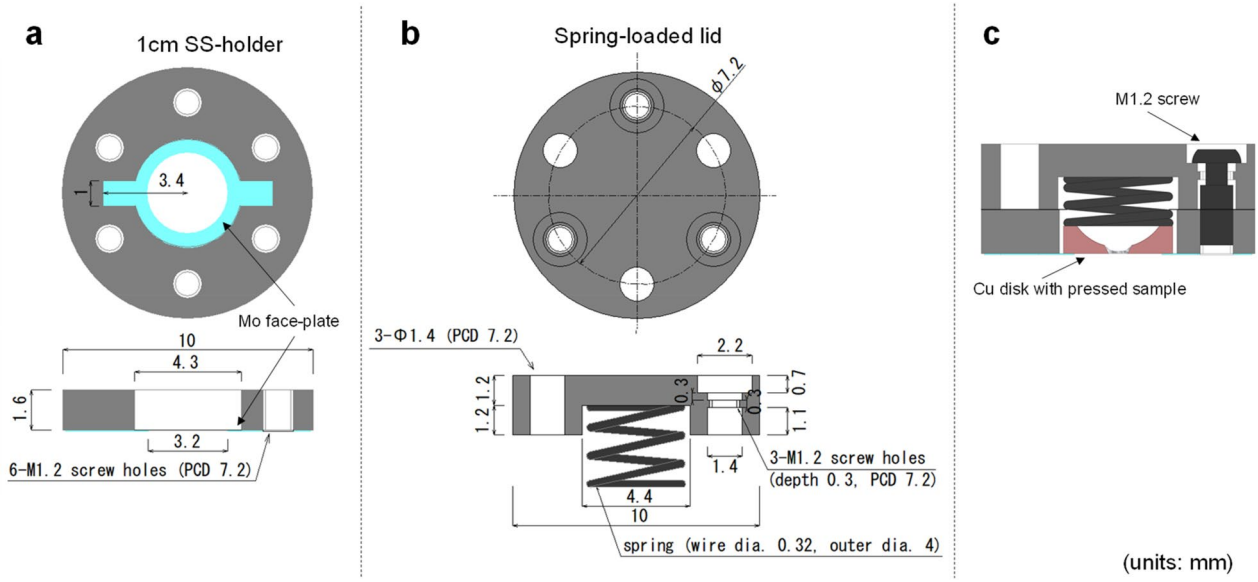


Fig. 5 Schematic diagram of the stainless-steel (SS) holder that had an outer diameter of 1 cm and was named as 1 cm SS-holder. **a**. Main block with a thin (50 μm) tungsten (W) face plate. **b**. Spring-loaded lid with a spring coil. **c.** Cross-section image when the Cu disk was installed in the 1 cm SS-holder. The sample surface was adjusted to the 50 μm below the W face plate

ConFlat (CF) ultra-high vacuum flanges of 70 mm in diameter (called CF70 flange). The container (Additional file 3) has four dimples to store the 1 cm SS-holders inside (Fig. 7a and b) and one screw hole for the fixing plate (Fig. 7c) that can be tightened with one screw (Fig. 7d). When grain samples (i.e., not mounted in the 1 cm SS-holder) were transported, samples were encapsulated in metal cups in advance using the sample encapsulation tool (described in the following subsection) and installed in the dimples. For the grain samples in metal cups, a different type of fixing disk (just a flat lid disk, without the springs, to prevent samples crushing) was used to cover the dimples. The CF70 flange-made transportation container was sealed with another CF70 flange using six M6 bolts (Fig. 7e) with a torque of 5 N (minimum of the standard torque). The screw part of the M6 bolts was wrapped with polytetrafluoroethylene sealing tape, which reduced the possibility of the cleaned bolts getting stuck in the screw holes.

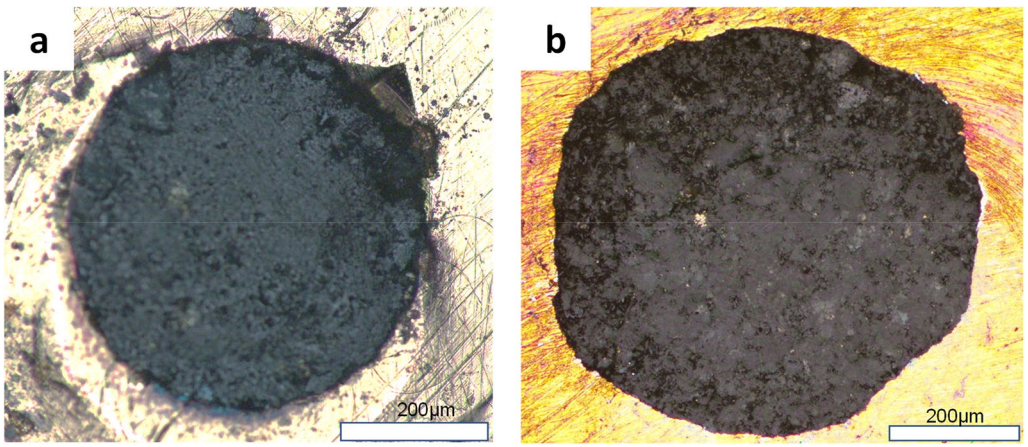


Fig. 6 Optical images of the pelletized Murchison samples. **a**. A Murchison pellet made of ~ 0.1 mg Murchison powder. **b**. A Murchison pellet made of ~ 0.3 mg Murchison powder

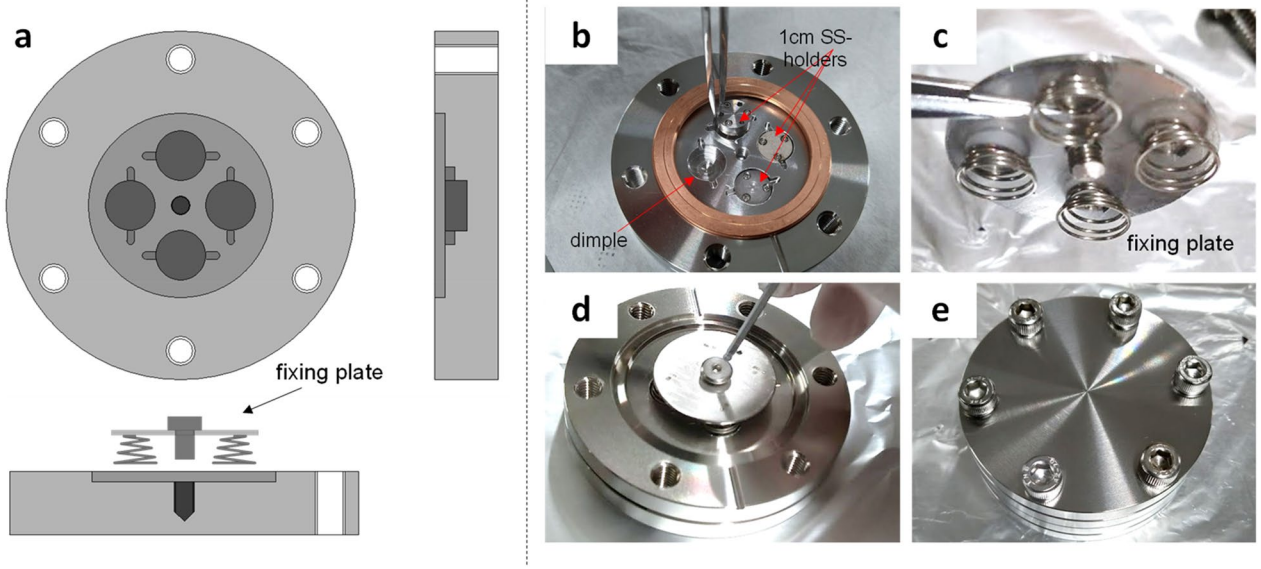


Fig. 7 Schematic diagram and photographs of the transportation container. **a**. The container was made from a ConFlat (CF) vacuum flange. **b**. Four × 1 cm SS- holders can be stored in the dimples. **c**. The 1 cm SS-holders were fixed using the fixing plate. **d**. The fixing plate can be fastened with one hand, **e**. The container was sealed using the other CF flange and a Cu metal gasket with a torque of 5 N

(D) FTIR/FESEM adapter holder and CaF_2 viewport

In order to carry out FTIR and FESEM analyses, the pelletized samples were transported to PMSCF, and were installed into holders specific to each instrument for these analyses. Because this installation was done in the glove box, repeated handling and exchange of individual 1 cm SS-holders would increase the risk of sample dropping, damage, and other issues. To reduce this risk, an adapter holder capable of handling multiple samples simultaneously was prepared. In addition, a CaF_2 viewport was developed for the FTIR that was airtight and able to contain the adapter holder. The adapter holder and the viewport were required to satisfy the instrumental constraints on the focal length of FESEM and FTIR, respectively.

The adapter holder (Fig. 8 and Additional file 4) was designed to contain three 1 cm SS-holders, and its outer diameter and height were suitable for the airtight FESEM holder that was developed and is routinely used at PMSCF. (The specifications and diagram of the airtight FESEM holder are confidential). The adapter (Table 1) holder was mounted on the airtight FESEM holder in the glove box, and introduced together into the FESEM for the observation.

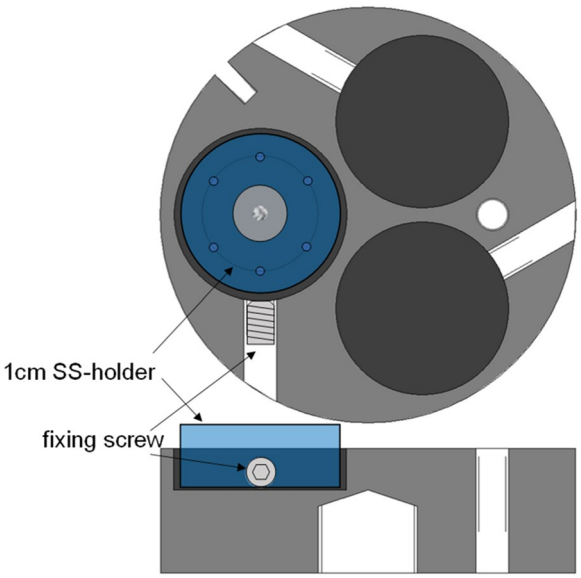


Fig. 8 Schematic diagram of the adapter holder for FESEM and FTIR analyses. Three 1 cm SS-holders can be installed in one adapter holder, and be fasten individually using fixing screws

The CaF_2 viewport for FTIR can contain one adapter holder (Fig. 9 and Additional file 5). The CaF_2 glass, 25 mm in diameter and 0.7 mm in thickness, was fixed with an O-ring. (The outer diameter of the O-ring is 24.5 mm, and the wire diameter is 1.5 mm.). The effective opening diameter of the CaF_2 viewport is 21 mm, which was large enough to observe the sample surface sideways visually for focusing during the FTIR analysis. The thickness (0.7 mm) of the CaF_2 glass had

Table 1 List of the sample-intimate components

Operation	Tool	Component	Material	Contact with
1. Sample collection	–	Tweezer, spatula	Stainless steel	Pristine sample
2. Pelletization	Tool B	Diamond disk	Diamond	Pristine sample
2. Pelletization	Tool B	Cu disk	Cu	Pristine sample
2. Pelletization	Tool B	Metal bead	Stainless steel	Pristine sample
4. Weighing	Tool E	Sample-pushing needle	Stainless steel	Pristine sample
4. Weighing	Tool E	Metal cup	Pt or Al	Pristine sample
7. NAA, 8. Ar–Ar dating	Tool G	Sapphire disk and lid	Sapphire	Pt-Os coated sample
7. NAA, 8. Ar–Ar dating	–	Spatula	Stainless steel	Pt-Os coated sample

"Pristine sample" means samples that remained in the pure nitrogen environment. "Pt-Os coated sample" means samples that were coated with Pt and Os for the SIMS analysis and handled under the Earth's atmosphere environment

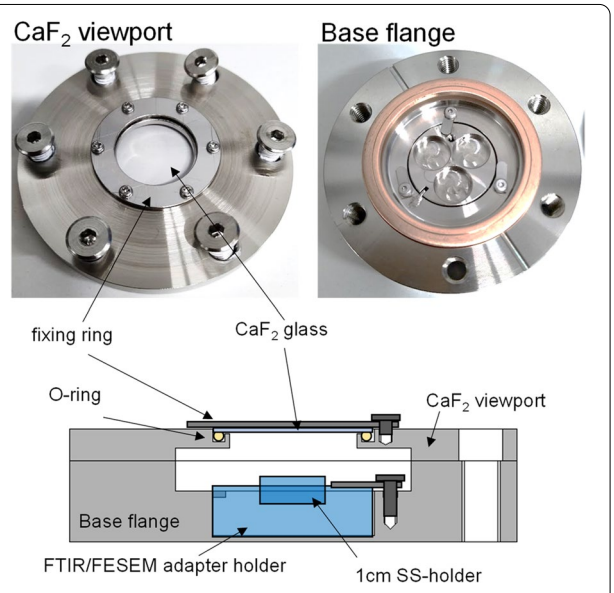


Fig. 9 Schematic diagram and photographs of the CaF_2 viewport, which was made from a ConFlat flange. The CaF_2 window is 25 mm in diameter, and the effective diameter is 20 mm

little effect on the FTIR spectroscopy (Okazaki et al. 2022).

(E) Sample encapsulation tool

After the FTIR and FESEM observations, mass spectrometry for noble gas, nitrogen, and volatile compounds was performed. These destructive analyses required that samples were encapsulated in appropriate cups for weighing and introduction into the instruments. When handling sub-millimeter sized samples in the glove box, it was necessary to prepare specific tools for safety and minimum contamination during the encapsulation of samples into metal cups. Also, the process of closing the metal cups had a risk of sample losses accompanied by deformation of the cups. Therefore, we prepared a tool to facilitate introduction of the pelletized sample into a metal (Al-, Ag-, or Pt-made) cup and sealing of the cap (Fig. 10 and Additional file 6).

The encapsulation tool was designed for a small metal cup (~2.5 mm in diameter and ~5 mm in height) that is usually used for noble gas analysis at Kyushu University. During the encapsulation operation, the metal cup was set in the supporting holder, and its height was adjusted to reduce the dead space between the cup and the Cu disk by using the cup-height adjusting screw (Fig. 10a and b). The Cu disk with the pelletized sample was placed above the metal cup and fixed to the cup supporting holder using the disk fixing plate (Fig. 10c). The pelletized sample in the Cu disk was pushed forward and dropped into the metal cup by using the sample-pushing needle (Fig. 10d). Finally, the cup was closed using the cup-sealing plate (Fig. 10d) and removed from the cup supporting holder.

For the encapsulation of a grain sample (not the pelletized sample), the disk fixing plate was replaced by a different plate with the conical hole that acted as a funnel in introducing the sample into the metal cup. When the grain sample was collected from the JAXA sapphire dish, the cup supporting holder was placed on the handling stage for the metal-cup holder (Fig. 2a and c).

(F) Weighing container

One of the simplest ways to measure the weight of samples without Earth's atmosphere exposure is to install and use a micro-balance in a glove box filled with pure nitrogen gas. This has an advantage regarding simplicity in handling samples. However, static electricity, fluctuation of the nitrogen pressure, and vibration of the glove box have problematic influences on the precision of weighing. Hence, a small container was developed for weighing samples with sub-milligram weight. The container should

be airtight, easy to handle in a glove box, and as lightweight as possible.

The weighing container and its lid (Fig. 11a and b, and Additional file 7) were made of aluminum alloy (Unified Numbering System (UNS): A96061). The lid was equipped with an elastomeric (JIS: B2410, ISO: 3601-5) an O-ring (Its outer diameter is 3.0 mm, and the wire diameter is 0.5 mm.) (Fig. 11a), which provided atmosphere-sealing performance when coupled with the container (Fig. 11c). The total blank weight of the container including the lid and the O-ring is ~ 130 mg, small enough to be measurable with a micro-balance (e.g., Sartorius MSU2.7S000DM and Mettler Toledo XPR2U that have a weighing capacity of 2100 mg and a 0.1 μg readability).

The weighing process was simple but repetitive as follows: (1) The total blank weight of the lid and the container (without coupling) was measured under the Earth's atmosphere environment, prior to the sample weighing. (2) The container and lid were introduced into the glove box. (3) The weighing container was set in the chase of the base plate (Fig. 11c), which prevented the container from rotating. (4) The sample encapsulated in the metal cup (the weight of the blank cup was measured in advance) was placed into the weighing container in the glove box. (5) The lid was coupled with the container and sealed by rotating with the lid-fastening rod (Fig. 11c) in the glove box. (6) The container with the sample was brought out from the glove box, and its weight was measured. The next sample weighing started from Step 1, because the blank weight could have changed due to a small fragment (~ 0.1 μg) generated from the container or the lid by closing/opening the lid.

In the rehearsal, we compared the weights of the blank container closed in Earth's atmosphere and that closed in the glove box repeatedly. This precaution test confirmed that any difference in the density between Earth's atmosphere and the nitrogen in the glove box did not affect their weights. The uncertainty of weighing using this weighing container and the micro-balance was about ± 0.2 μg on average of 5 to 7 measurements for each sample.

(G) Sapphire container for samples handled in Earth's atmosphere

A different container was prepared to transport a single grain or fine-grained natural and standard samples for

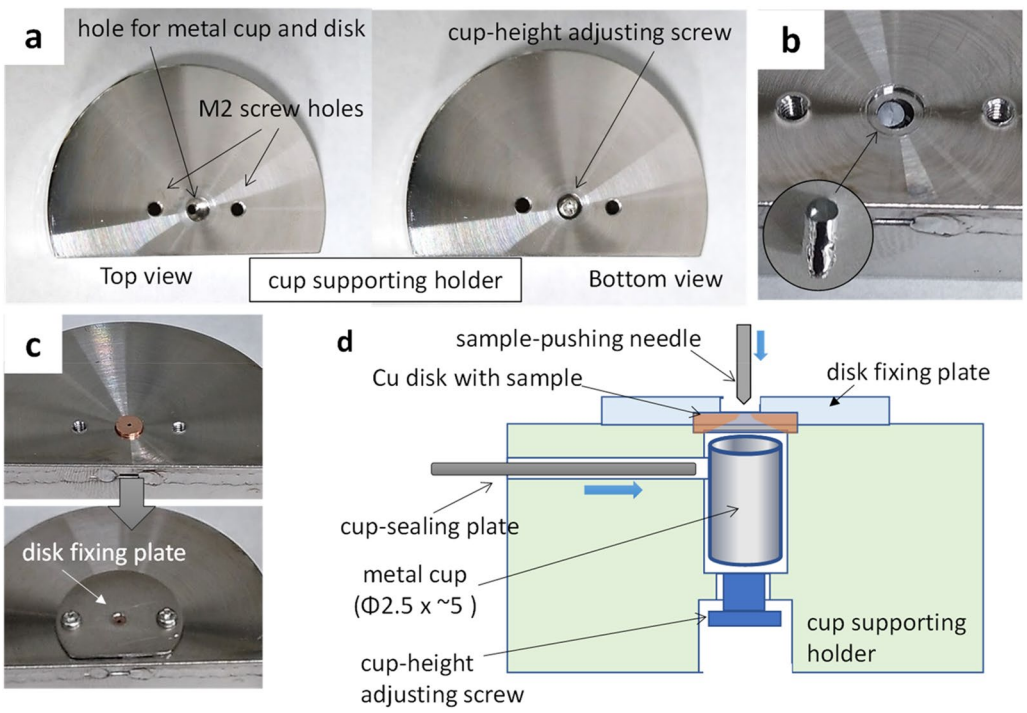


Fig. 10 Photographs and a schematic diagram of the sample encapsulation tool. **a**. The cup supporting holder had a hole for the metal cup and Cu disk. The height of the cup can be adjusted with a cup-height adjusting screw. **b**. A metal cup of < 3 mm diameter can be installed. **c**. A Cu disk with a pelletized sample was set and fastened with the cup supporting holder using the disk fixing plate. **d**. The pelletized sample was dropped off from the Cu disk by pushing it forward with the sample-pushing needle (made of stainless-steel), and then the metal cup was closed by pressing it with the cup-sealing plate

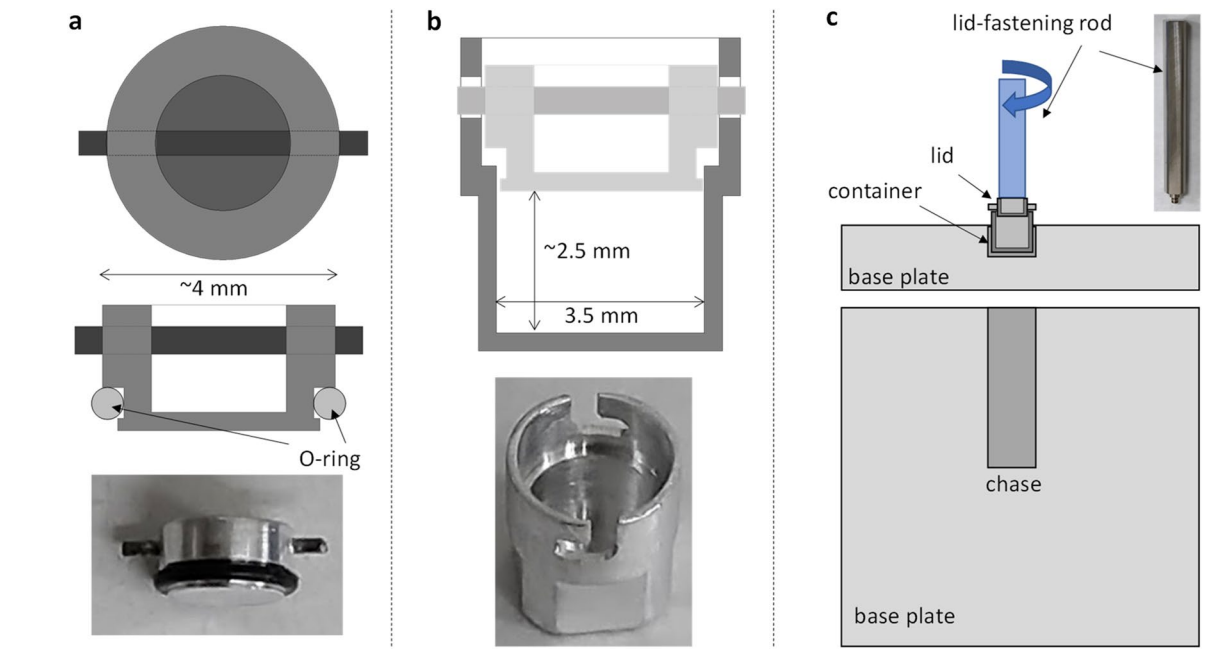


Fig. 11 Schematic diagrams and photographs of the weighing container. **a**. The lid has an elastomeric O ring. **b**. The container has an inner space of 3.5 mm in diameter and ~ 2.5 mm in height. **c**. The container can be fasten with one hand by using the base plate and the lid-fastening rod

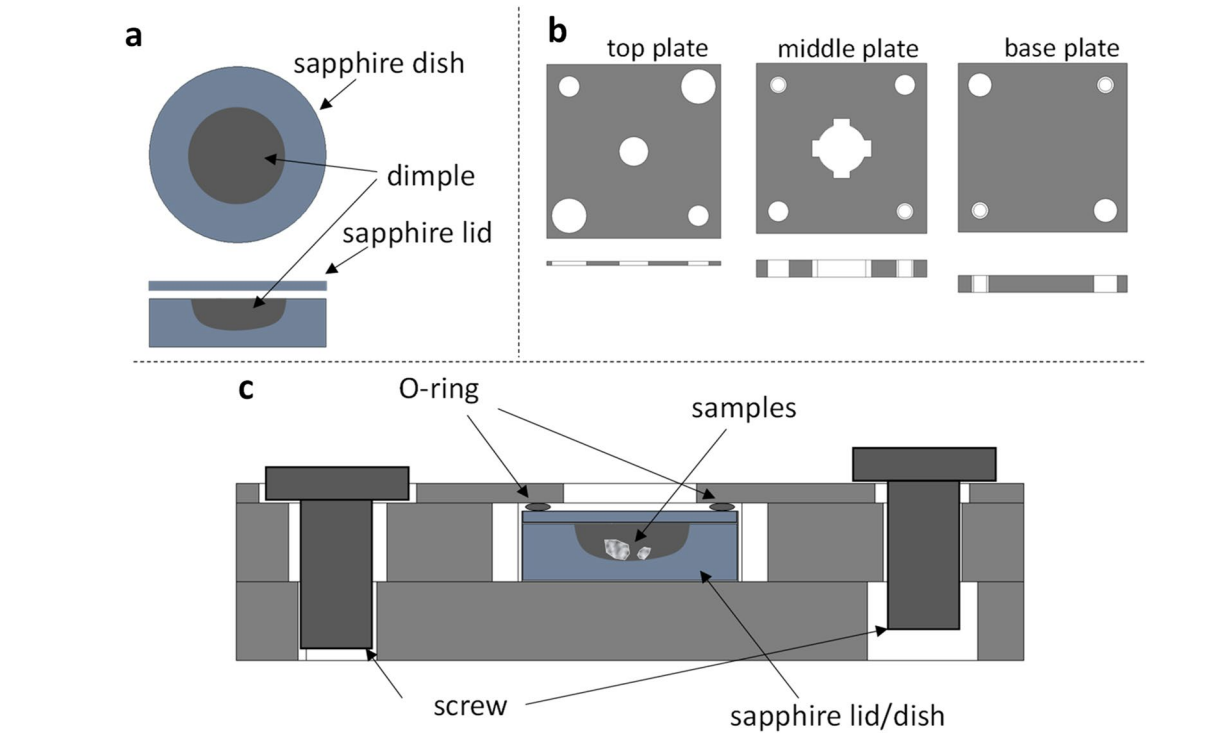


Fig. 12 Schematic diagram of the sapphire container and the metal holder. **a**. The sapphire dish is 5.5 mm in diameter and 1.5 mm in thickness. The dimple was made by a portable grinder and polished diamond powder. **b**. The metal holder consists of three parts made of stainless steel, which are easily washed and assembled. **c.** The samples placed in the sapphire dish were enclosed with the lid and the O-ring

the NAA measurement (Fig. 1). This container was not required to be airtight, but had the following requirements: (1) The container must be easy to handle when taking samples in and out. (2) The container must be chemically clean. (3) The samples packed in the container must be visible with the lid on, when handling powders or small particles, which are greatly affected by static electricity, it is important to observe them before opening the lid. (4) The container must have a simple structure to be easily mass-produced.

The container (Additional file 8) consists of a sapphire lid and a sapphire dish (Fig. 12a), and a holder made of stainless steel (Fig. 12b). The sapphire dish is 5.5 mm in diameter and 1.5 mm in thickness and has a dimple that was made by a portable grinder to fit the dimple size with each of the samples. The maximum sample size appreciable is ~ 1 mm. After the grinding, the dimple was polished with diamond powders (9 μm and 15 μm in diameter respectively). The lid is 5.5 mm in diameter and 0.3 mm in thickness, and translucent to view the samples. The sapphire dishes and lids, stainless-steel holders, and screws were washed sequentially with neutral detergent in hot water, acetone, ethanol, and pure water using an ultrasonic cleaner. The washed dishes and lids were then heated at 220 °C in a vacuum for 11 h. No residues or deposits larger than about 10 μm in size were observed on the dishes or lids under a microscope. The elastomeric (JIS: B2410, ISO: 3601-5) O-ring was washed with ethanol and pure water, and then dried in a vacuum at room temperature.

Figure 12c depicts the state in which the container stored the sample. The first step in the process of storing the sample was to assemble the holder and the sapphire dish: the mid plate and the base plate of the stainless-steel holder were combined with two screws (the left screw in Fig. 12c), and the sapphire dish was set in the center hole of the middle plate. Then, a sample was placed in the dimple of the dish. The lid was put on the dish and closed by pressing together an O-ring (4.0 mm in outer diameter and with a 0.5 mm wire diameter) and the top plate by fastening the other two screws (the right screw in Fig. 12c). The installed sample could be seen through the center hole of the top plate. We did not experience any samples escaping due to an accidental loosening of the lid.

Summary

We have developed the tools for handling, transportation, weighing, and pelletization, which were applied to the initial analysis of volatile components in the Hayabusa2 sample. Many improvements and modifications enabled us to process and handle the samples successfully under non-atmospheric exposure conditions during transportation, weighing, pelletization, and installation into the measurement instruments. All the tools presented here will hopefully be modified and used in future analyses of small samples.

Abbreviations

PMSCF: Planetary Material Sample Curation Facility; ISAS: Institute of Space and Astronautical Science; JAXA: Japan Aerospace Exploration Agency; FTIR: Fourier Transfer Infrared Spectroscopy; FESEM: Field Emission Scanning Electron Microscope; SIMS: Secondary-Ion Mass Spectrometry; NAA: Neutron Activation Analysis; STP: Standard Temperature and Pressure; CF: ConFlat.

Supplementary Information

The online version contains supplementary material available at https://doi.org/10.1186/s40623-022-01747-7.

Additional file 1: Fig. S1 Draws of the handling stage.

Additional file 2: Fig. S2 Draws of the pelletization tool.

Additional file 3: Fig. S3 Draws of the transportation container.

Additional file 4: Fig. S4 Draws of the adapter holder for FESEM and FTIR.

Additional file 5: Fig. S5 Draws of the CaF_2 viewport.

Additional file 6: Fig. S6 Draws of the sample encapsulation tool.

Additional file 7: Fig. S7 Draws of the weighing container.

Additional file 8: Fig. S8 Draws of the metal holder for the sapphire container.

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Author contributions

RO led and planned the analysis and prepared the manuscript together with TY and FK. RO, SY, and AB designed and developed all of the tools. KS and RO evaluated the pelletized sample surface flatness. TY and FK tested the tools for FESEM and FTIR observations. All authors read and approved the final manuscript.

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Availability of data and materials

The data used in this study are available from the corresponding author on reasonable request.

Declarations

Ethics approval and consent to participate

Not applicable.

Consent for publication

Not applicable.

Competing interests

The authors declare that they have no competing interests.

Author details

[1]Department of Earth and Planetary Sciences, Faculty of Sciences, Kyushu University, 744 Motooka, Nishi-Ku, Fukuoka 819-0395, Japan. [2]Research Equipment Development Center of Science Faculty, Kyushu University,

744 Motooka, Nishi-Ku, Fukuoka 819-0395, Japan. [3]Department of Earth and Planetary Sciences, School of Science, Kyushu University, 744 Motooka, Nishi-Ku, Fukuoka 819-0395, Japan. [4]Institute of Space and Astronautical Science, Japan Aerospace Exploration Agency, Sagamihara, Japan.

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References

Bland PA, Zolensky ME, Benedix GK, Sephton MA (2006) Weathering of chondritic meteorites. In: Lauretta DS, McSween HY Jr (eds) Meteorites and the early solar system II. University of Arizona Press, Tucson, pp 853–867

Britt DT, Yeomans D, Housen K, Consolmagno G (2002) Asteroidal density, porosity, and structure. In: Bottke WF, Cellino A, Paolicchi P, Binzel RP (eds) Asteroids III. University of Arizona Press, Tucson, pp 485–500

King AJ, Phillips KJH, Strekopytov S, Vita-Finzi C, Russell SS (2020) Terrestrial modification of the Ivuna meteorite and a reassessment of the chemical composition of the CI type specimen. Geochim Cosmochim Acta 268:73–89. https://doi.org/10.1016/j.gca.2019.09.041

Mazor E, Heymann D, Anders E (1970) Noble gases in carbonaceous chondrites. Geochim Cosmochim Acta 34:781–824. https://doi.org/10.1016/0016-7037(70)90031-1

Okazaki R, Marty B, Busemann H, Hashizume K, Gilmour J, Meshik A, Yada T, Kitajima F, Broadley M, Byrne D, Füri E, Riebe MEI, Krietsch D, Maden C, Ishida A, Clay P, Crowther SA, Fawcett L, Lawton T, Pravdivsteva O, Miura YN, Park J, Bajo K, Takano Y, Yamada K, Kawagucci S, Matsui Y, Yamamoto M, Righter K, Sakai S, Iwata N, Shirai N, Sekimoto S, Inagaki M, Ebihara M, Yokochi R, Nishiizumi K, Nagao K, Lee JI, Kano A, Caffee MW, Uemura R, Nakamura T, Naraoka H, Noguchi T, Yabuta H, Yurimoto H, Tachibana S, Sawada H, Sakamoto K, Abe M, Arakawa M, Fujii A, Hayakawa M, Hirata N, Hirata N, Honda R, Honda C, Hosoda S, Iijima Y, Ikeda H, Ishiguro M, Ishihara Y, Iwata T, Kawahara K, Kikuchi S, Kitazato K, Matsumoto K, Matsuoka M, Michikami T, Mimasu Y, Miura A, Morota T, Nakazawa S, Namiki N, Noda H, Noguchi R, Ogawa N, Ogawa K, Okada T, Okamoto C, Ono G, Ozaki M, Saiki T, Sakatani N, Senshu H, Shimaki Y, Shirai K, Sugita S, Yakei Y, Takeuchi H, Tanaka S, Tatsumi E, Terui F, Tsukizaki R, Wada K, Yamada M, Yamada T, Yamamoto Y, Yano H, Yokota Y, Yoshihara K, Yoshikawa M, Yoshikawa K, Furuya S, Hatakeda K, Hayashi T, Hitomi Y, Kumagai K, Miyazaki A, Nakato A, Nishimura M, Soejima H, Iwamae A, Yamamoto D, Yogata K, Yoshitake M, Fukai R, Usui T, Connolly HC Jr, Lauretta D, Watanabe S, Tsuda Y (2022) Noble gases and nitrogen in samples of asteroid Ryugu record its volatile sources and recent surface evolution. Science. https://doi.org/10.1126/science.abo0431

Okazaki R, Marty B, Busemann H, Miura YN, Yamada K, Sakai S, Hashizume K, Bajo K, Sekimoto S, Kitajima F, Righter K, Meshik A, Gilmour JD, Iwata N, Füri E, Crowther SA, Shirai N, Ebihara M, Takano Y, Ishida A, Yokochi R, Pravdivtseva O, Park J, Yada T, Nishiizumi K, Nagao K, Lee JI, Broadley MW, Byrne D, Riebe MEI, Clay P, Kano A, Caffee M, Kawagucci S, Matsui Y, Uemura R, Inagaki M, Krietsch D, Maden C, Yamamoto M, Yurimoto H, Nakamura T, Noguchi T, Naraoka H, Yabuta H, Sakamoto K, Tachibana S, Watanabe S, Tsuda Y (2021) Initial analysis of volatile components in the Hayabusa2 samples. Hayabusa Symposium 2021: #S3–1. https://curation.isas.jaxa.jp/symposium/abstract/2021/Abstracts_all.pdf

Tachibana S, Abe M, Arakawa M, Fujimoto M, Iijima Y, Ishiguro M, Kitazato K, Kobayashi N, Namiki N, Okada T, Okazaki R, Sawada H, Sugita S, Takano Y, Tanaka S, Watanabe S, Yoshikawa M, Kuninaka H, The Hayabusa2 project team (2014) Hayabusa2: Scientific importance of samples returned from C-type near-Earth asteroid (162173) 1999 JU_3. Geochem J 48:571–587. https://doi.org/10.2343/geochemj.2.0350

Tachibana S, Sawada H, Okazaki R, Takano Y, Sakamoto K, Miura YN, Okamoto C, Yano H, Yamanouchi S, Michel P, Zhang Y, Schwartz S, Thuillet F, Yurimoto H, Nakamura T, Noguchi T, Yabuta H, Naraoka H, Tsuchiyama A, Imae N, Kurosawa K, Nakamura AM, Ogawa K, Sugita S, Morota T, Honda R, Kameda S, Tatsumi E, Cho Y, Yoshioka K, Yokota Y, Hayakawa M, Matsuoka M, Sakatani N, Yamada M, Kouyama T, Suzuki H, Honda C, Yoshimitsu T, Kubota T, Demura H, Yada T, Nishimura M, Yogata K, Nakato A, Yoshitake M, Suzuki AI, Furuya S, Hatakeda K, Miyazaki A, Kumagai K, Okada T, Abe M, Usui T, Ireland TR, Fujimoto M, Yamada T, Arakawa M, Connolly HC Jr, Fujii A, Hasegawa S, Hirata N, Hirata N, Hirose C, Hosoda S, Iijima Y, Ikeda H, Ishiguro M, Ishihara Y, Iwata T, Kikuchi S, Kitazato K, Lauretta DS, Libourel G, Marty B, Matsumoto K, Michikami T, Mimasu Y, Miura A, Mori O, Nakamura-Messenger K, Namiki N, Nguyen AN, Nittler LR, Noda H, Noguchi R, Ogawa N, Ono G, Ozaki M, Senshu H, Shimada T, Shimaki T, Shirai K, Soldini S, Takahashi T, Takei Y, Takeuchi H, Tsukizaki R, Wada K, Yamamoto Y, Yoshikawa K, Yumoto K, Zolensky ME, Nakazawa S, Terui F, Tanaka S, Saiki T, Yoshikawa M, Watanabe S, Tsuda Y (2022) Pebbles and sand on asteroid (162173) Ryugu: in situ observation and particles returned to Earth. Science 375:1011–1016. https://doi.org/10.1126/science.abj8624

Thomen A, Remusat L, Robert F, Meibom A, Mostefaoui S (2010) Chemical and nitrogen isotopic composition of the *hotspots* in Orgueil insoluble organic matter. 41st Lunar Planet Sci Conf, #2472. https://www.lpi.usra.edu/meetings/lpsc2010/pdf/2472.pdf

Yada T, Abe M, Okada T, Nakato A, Yogata K, Miyazaki A, Hatakeda K, Kumagai K, Nishimura M, Hitomi Y, Soejima H, Yoshitake M, Iwamae A, Furuya S, Uesugi M, Karouji Y, Usui T, Hayashi T, Yamamoto D, Fukai R, Sugita S, Cho Y, Yumoto K, Yabe Y, Bibring JP, Pilorget C, Hamm V, Brunetto R, Riu L, Lourit L, Loizeau D, Lequertier G, Moussi-Soffys A, Tachibana S, Sawada H, Okazaki R, Takano Y, Sakamoto K, Miura YN, Yano H, Ireland TR, Yamada T, Fujimoto M, Kitazato K, Namiki N, Arakawa M, Hirata N, Yurimoto H, Nakamura T, Noguchi T, Yabuta H, Naraoka H, Ito M, Nakamura E, Uesugi K, Kobayashi K, Michikami T, Kikuchi H, Hirata N, Ishihara Y, Matsumoto K, Noda H, Noguchi R, Shimaki Y, Shirai K, Ogawa K, Wada K, Senshu H, Yamamoto Y, Morota T, Honda R, Honda C, Yokota Y, Matsuoka M, Sakatani N, Tatsumi E, Miura A, Yamada M, Fujii A, Hirose C, Hosoda S, Ikeda H, Iwata T, Kikuchi S, Mimasu Y, Mori O, Ogawa N, Ono G, Shimada T, Soldini S, Takahashi T, Takei Y, Takeuchi H, Tsukizaki R, Yoshikawa K, Terui F, Nakazawa S, Tanaka S, Saiki T, Yoshikawa M, Watanabe S, Tsuda Y (2022) Preliminary analysis of the Hayabusa2 samples returned from C-type asteroid Ryugu. Nat Astron 6:214–220. https://doi.org/10.1038/s41550-021-01550-6

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