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Technical report

Development of a new sample holder and sample holder container for coordinated surface analyses (micro-IR, XPS, FE-SEM, and micro-Raman) and ion irradiation experiments of extraterrestrial materials

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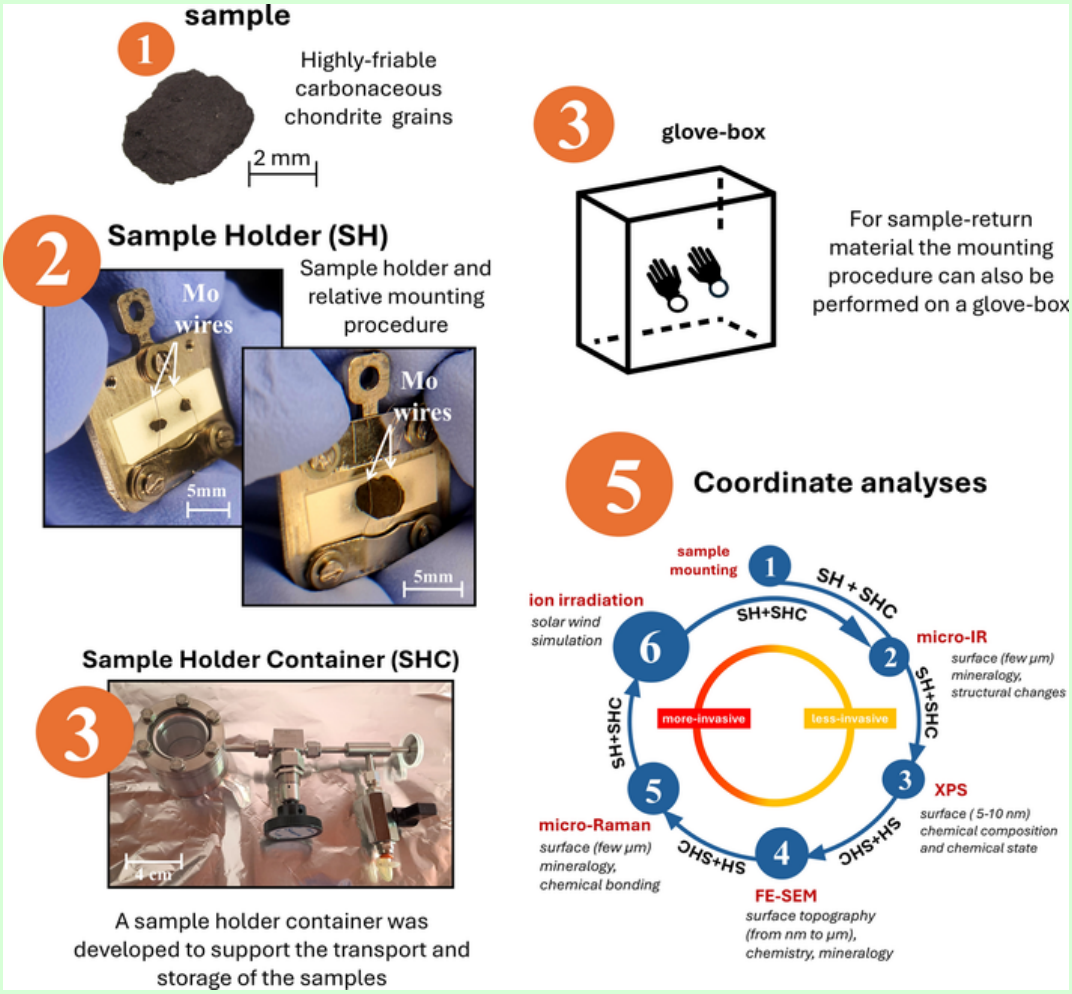
Abstract

The Hayabusa2 and OSIRIS-REx missions have successfully returned pristine materials from the **AQ1** carbonaceous asteroids Ryugu and Bennu, respectively. These missions offer a unique opportunity to study space weathering processes, primarily driven by solar wind irradiation and micrometeorite bombardment, which continuously affect the surface of airless bodies. Coordinated surface and sub-surface techniques, including micro-infrared spectroscopy (micro-IR), X-ray photoelectron spectroscopy (XPS), field emission scanning electron microscopy (FE-SEM), and micro-Raman spectroscopy, have proven particularly valuable for unveiling the nature of these intricate processes. However, a sample holder specifically designed for extraterrestrial samples to facilitate coordinated surface analyses has yet to be reported. This study presents the development and application of a new sample holder (SH) and sample holder container (SHC), specifically designed to optimize the efficiency of coordinated surface analyses of extraterrestrial materials while preserving their integrity. The SH securely holds irregular millimeter-sized, high-friable grains (e.g., Ivuna-type carbonaceous chondrites) for sequential analyses such as micro-IR, XPS, FE-SEM, micro-Raman, and ion irradiation. The latter is critical for simulating space weathering processes. The SH was designed with high-purity materials, including molybdenum and alumina (Al_2O_3), ensuring low chemical contamination risk and high-mechanical stability. The SHC complements this setup by providing a secure solution for transporting samples among different facilities. It allow to maintain an inert gas environment or high vacuum condition to prevent contamination from exposure to the terrestrial atmosphere. This combined system was successfully applied in the coordinated surface analysis of two Ryugu grains, preserving their chemical and physical integrity while facilitating detailed investigations into space weathering effects. These technical advancements provide a robust framework for multidisciplinary research on sensitive extraterrestrial materials, ensuring accurate and precise results and securing sample integrity throughout the coordinated analyses.

Graphical Abstract



Keywords

Sample holder
Mounting procedures
Extraterrestrial materials
Surface analyses
Ryugu returned grains

Abbreviations

ASRG Astromaterials Science Research Group
CI Ivuna-type carbonaceous chondrite
EDS Energy dispersive spectroscopy
FE-SEM Field emission scanning electron microscope
HV High vacuum
IR Infrared
JAXA Japan Aerospace Exploration Agency
Mo-FSP Molybdenum flag-style plate
SH Sample holder
SHC Sample holder container
SXF X-ray fluorescence
UHV Ultra-high vacuum
Ta-s1 Tantalum screws 1
Ta-s2 Tantalum screws 2
TFCT Tweezer with fine-cut tips
XPS X-ray photoelectron spectroscopy

1. Introduction

Hayabusa2 and OSIRIS-REx are pioneering sample-return missions that successfully collected pristine materials from the primitive carbonaceous asteroids (162173) Ryugu ~~(162173)~~ and (101955) Bennu ~~(101955)~~, respectively (e.g., Ito et al. 2022; Nakamura et al. 2022; Yokoyama et al. 2023; Lauretta et al. 2024). These missions aim to deepen our understanding of the Solar System and planetary formation by enabling the investigation of primordial rocky materials in advanced Earth-based research laboratories (e.g., Watanabe et al. 2017; Lauretta et al. 2019, 2024; Ito et al. 2022; Nakamura et al. 2022). These missions offer a unique opportunity to study the chemical and physical alterations induced by space weathering processes (e.g., micrometeorite impacts and solar wind irradiation), which continuously modify the surface of airless bodies (Thompson et al. 2016; Noguchi et al. 2011, 2023). As demonstrated by laboratory experiments, space weathering is a complex research field as these processes occur through diverse chemical and physical mechanisms at varying depths, from nanometers to microns (Sasaki et al. 2001; Loeffler et al. 2009; Laczniak et al. 2021; Chaves and Thompson 2022). Thus, coordinated surface and sub-surface analytical approaches are essential to gain insights into these intricate phenomena (e.g., Laczniak et al. 2021). Techniques such as infrared spectroscopy (micro-IR), X-ray photoelectron spectroscopy (XPS), field emission scanning electron microscopy (FE-SEM), and micro-Raman spectroscopy have proven to be particularly valuable (Dukes et al. 1999; López-Oquendo et al. 2024; Bott et al. 2024; Rubino et al. 2024a). These methods enable detailed analyses of mineralogy, structural modifications, surface topography, and chemical changes, providing key findings into the complex **AQ2** interactions driven by those processes.

Various sample holder and mounting solutions have been proposed for techniques such as micro-IR, XPS, FE-SEM, micro-Raman, and ion irradiation. For instance, slice samples can be mechanically fixed on the sample holder (Laczniak et al. 2021), while irregular shape and/or powder samples can be attached on a double-sided carbon tape, indium plate, or compressed in the shape of a pellet (e.g., Loeffler et al. 2009; Stevie et al. 2020; Brunetto et al. 2020).

It is important to note that, given the great scientific impact of extraterrestrial materials such as returned samples, all analytical procedures must be accurately defined before any investigative study in order to minimize terrestrial contamination. For this reason, extensive research has been conducted on their sampling, preservation, manipulation, and analysis (e.g., Brownlee et al. 2006; Allen et al. 2011; Allen 2012; Okazaki et al. 2017; McCubbin et al. 2019; Ito et al. 2020; Aléon-Toppani et al. 2021; Yada et al. 2023). Sample preparation, handling, and mounting are crucial aspects of many analytical techniques (e.g., Kebukawa et al. 2009; Stevie et al. 2020; Wilson et al. 2024). During these procedures, material loss and organic and inorganic chemical contamination from the analytical instrumentations can occur, especially with highly friable samples like those from Ryugu and Bennu (e.g., Kebukawa et al. 2009; Shirai et al. 2020; Yokoyama et al. 2023; Lauretta et al. 2024), which are similar to Ivuna-type (CI-type) carbonaceous chondrites (e.g., Blinova et al., 2014 2024). Therefore, numerous strategies have been proposed in the literature to address these challenges when analyzing returned samples using both non-destructive and destructive (e.g., Ito et al. 2022; Nakamura et al. 2022; Rubino et al. 2023; Yokoyama et al. 2023) techniques. For example, specific sample holders have been designed for IR spectroscopy, X-ray tomography (Uesugi et al. 2020), and coordinated nano-analyses (e.g., TEM, NanoSIMS, and STXM-NEXAFS) (e.g., Ito et al. 2020; Yamaguchi et al. 2023; Nakashima et al. 2023). Methods for preparing polished sections have also been developed for SEM analyses (e.g., Yamaguchi et al. 2023; Nakashima et al. 2023; Wilson et al. 2024), along with new solutions for the investigation of extraterrestrial materials collected from future Mars sample returned missions (Adam et al. 2024). However, a sample holder specifically designed for extraterrestrial samples and returned materials to facilitate coordinated surface analyses has yet to be documented in the literature.

In this work, we propose a new sample holder (SH) suitable for the analysis of irregular, high-friable, and mm-sized grains (1.5–10 mm)—e.g., CI-type and other similar carbonaceous chondrites—using coordinated surface and sub-surface techniques (micro-IR, XPS, FE-SEM, and micro-Raman) and ion irradiation processing to simulate the effects induced by solar particles. Moreover, a sample holder container (SHC) was developed to support the transport and storage of the SH in a secure and safe manner, thus minimizing the contamination of the terrestrial atmosphere. Finally, the SH and associated mounting procedure presented here were successfully applied to the investigation of two Ryugu grains allocated to our research team during the second Announcement of Opportunity by Astromaterials Science Research Group (ASRG) promoted by the Japan Aerospace Exploration Agency (JAXA) in June 2022 (Palomba et al. 2024; Shehaj et al. 2024; Rubino et al. 2024b).

2. Materials and methods

The sample mounting test and XPS analysis were performed in the MATerial CHaracterization LABoratory (MATCHLAB) of the Università degli Studi di Firenze, Italy. The Tagish Lake meteorite, a C2 ungrouped carbonaceous chondrite, has been used in our mounting tests as it closely resembles Ryugu and Bennu grains in terms of density and friability (Blinova et al. 2014; Yokoyama et al. 2023; Lauretta et al. 2024). Furthermore, the samples were used without any preparation or treatment.

The photoelectron spectra were acquired by a PREVAC standard PSE system equipped with a non-monochromatic X-ray source Mg K-alpha (1253.6 eV) and hemispherical electron energy analyzer EA15 operating in the constant pass energy mode of 44 eV. The photoelectrons were collected perpendicular to the sample surface. The pressure during the experiment was keep below 2×10^{-7} Pa. The calibration of spectra was obtained shifting to 284.8 eV the lowest component relative to the 1 s transition of the adventitious carbon (Biesinger 2022).

Infrared spectroscopy tests were conducted at the SINBAD beamline—DAFNE synchrotron (INFN-LNF Roma, Italy)—using the FR-IR LUMOS II (Bruker) infrared microscope. Ryugu samples were measured under a glovebox with N_2 enriched atmosphere to limit the terrestrial alteration of the samples. The surface of each grain was characterized in the mid-IR (2.5–16 µm) using the Lumos II FT-IR microscope, with × 8 magnification in reflectance mode using MCT-LN mapping (256 scans, 4 cm^{-1} spectral resolution, 80 µm aperture). The standard used is a gold plate.

The electron microscope measurements were conducted using a Zeiss EVO MA15 scanning electron microscope (SEM) equipped with a 40 mm^2 Silicon Drift Detector (SDD) and Oxford UltimMax and processed with Aztec 5.1 microanalysis software at the electron microscopy and microanalysis facility (MEMA) of the Università degli Studi di Firenze, Italy. Semiquantitative energy dispersive X-ray spectroscopy (EDS) analyses were carried out at 15 kV acceleration voltage and 700 pA nominal beam current, 6300 cps as average count rate, and acquisition livetime of 450 s, in high vacuum conditions ($\sim 10^{-3}$ Pa). Semiquantitative chemical data were obtained from point analyses by converting count rates to concentrations using energy and beam calibration on a pure cobalt standard and factory quantitative standardizations for the elements of interest. Elemental maps were acquired by EDS operating at 15 kV accelerating potential and 700 pA probe currents, 7200 cps as average count rate on the whole spectrum, and acquisition livetime of 630 s. Further test and measurements were performed using a Thermo Scientific Apreo Field Emission-Scanning Electron Microscopy at the Centre for Electron Microscopy, Eindhoven NanoPort (Netherlands). The high-resolution images were acquired by secondary electron mode in high-vacuum conditions ($\sim 10^{-3}$ Pa) with an accelerating voltage of 1 kV and a beam current of 6.3 pA.

Raman analysis were carried out using a Raman spectrometer Horiba Jobin–Yvon LabRam 300 coupled with an optical microscope Olympus BX41 at MEMA laboratory. The Raman device was equipped with a HeNe laser source ($\lambda_0 = 632.8$ nm), a monochromator with holographic edge filter, a spectrometer with diffraction grating of 1800 g/mm, and a Peltier cooled Horiba Odyssey CCD detector (1024 × 256 pixels) allowing a spectral resolution of about 1 cm^{-1}. To prevent thermal modification, the laser delivered a power of approximately 1mW at the sample surface. The beam was focused with a 50X long working distance objective (Olympus MS Plan NA 0.55). Spectra were calibrated at 520.5 cm^{-1} using a silicon wafer.

The ion bombardment tests were carried out at the Laboratory for Experimental Astrophysics of the Osservatorio Astrofisico di Catania, Italy. Ion irradiation is performed at room temperature in a vacuum chamber kept at a pressure in the range of 10^{-7} Pa. The samples were irradiated by Ar^{2+} ions at an energy of 400 keV and fluence of 1×10^{15} ions/cm^2 for the first irradiation and 3×10^{15} ions/cm^2 for the second, both performed using a 200 kV Danfysik implanter.

3. Requirements of the SH for coordinate surface analyses

To ensure a successful coordinated analysis of extraterrestrial returned samples, several key requirements must be satisfied to preserve their pristine state and facilitate accurate and precise measurements across various analytical techniques. The samples must be securely and safely transported in an SH compatible with different analytical instruments, such as micro-IR, XPS, FE-SEM, and micro-Raman.

The SH must be capable of holding the sample in different analytical environments, including Ultra-High Vacuum (UHV) conditions, within the range 10^{-7} to 10^{-8} Pa (e.g., XPS analysis), as well as at atmospheric pressure (e.g., for the mounting). Additionally, the SH should be designed to withstand ion bombardment during solar wind simulation experiments, securing sample integrity in such conditions (e.g., Ar^{2+} ions at an energy of 400 keV and fluence of 3×10^{15} ions/cm^2). The SH must hold irregular, high-friable grains (1.5–10 mm) stable and fixed to prevent drift motion during coordinate analyses while allowing the same surface to remain exposed for all measurements. Handling procedures should involve using clean tools to minimize contact with the region of interest as much as possible. Additionally, no conductive coating (e.g., graphite) or any other sample preparation should be applied to the sample surface to preserve its pristine condition, which is crucial for techniques such as XPS.

It is essential to minimize analytical contamination and terrestrial alteration as much as possible (e.gGounelle and Zolensky 2001; Kebukawa et al. 2009; Shirai et al. 2020; Imae et al. 2024), preserving the grains' original chemistry, texture, and morphology. The SH should also minimize shadow effects across various techniques, ensuring precise and accurate measurements. To streamline analyses, the grains must be easily mounted and removed. Moreover, a carefully designed analytical pipeline is essential to preserve the sample's integrity and minimize analytical artifacts introduced by instrumentation.

4. Requirements for the SHC and transportation of the SH

The SH must be transported securely among laboratories (facility-to-facility transfer), ensuring complete protection from exposure to the Earth's atmosphere. Such exposure could irreversibly alter the sample's pristine chemical and physical properties, compromising its integrity and suitability for the coordinate surface analyses (e.g., Loeffler et al. 2009; Jenkins et al. 2024). To achieve this goal, a transport container must be specifically designed to host the SH safely during transport and storage. The container should be compatible with the SH, easy to use under a controlled atmosphere (i.e., glove box), and robust enough to ensure seamless handling. To further mitigate the risk of contamination, the container should maintain a controlled internal environment, by operating under an inert protective atmosphere (e.g., nitrogen) or maintaining high-vacuum conditions (up to ~ $1 \cdot 10^{-5}$ Pa).

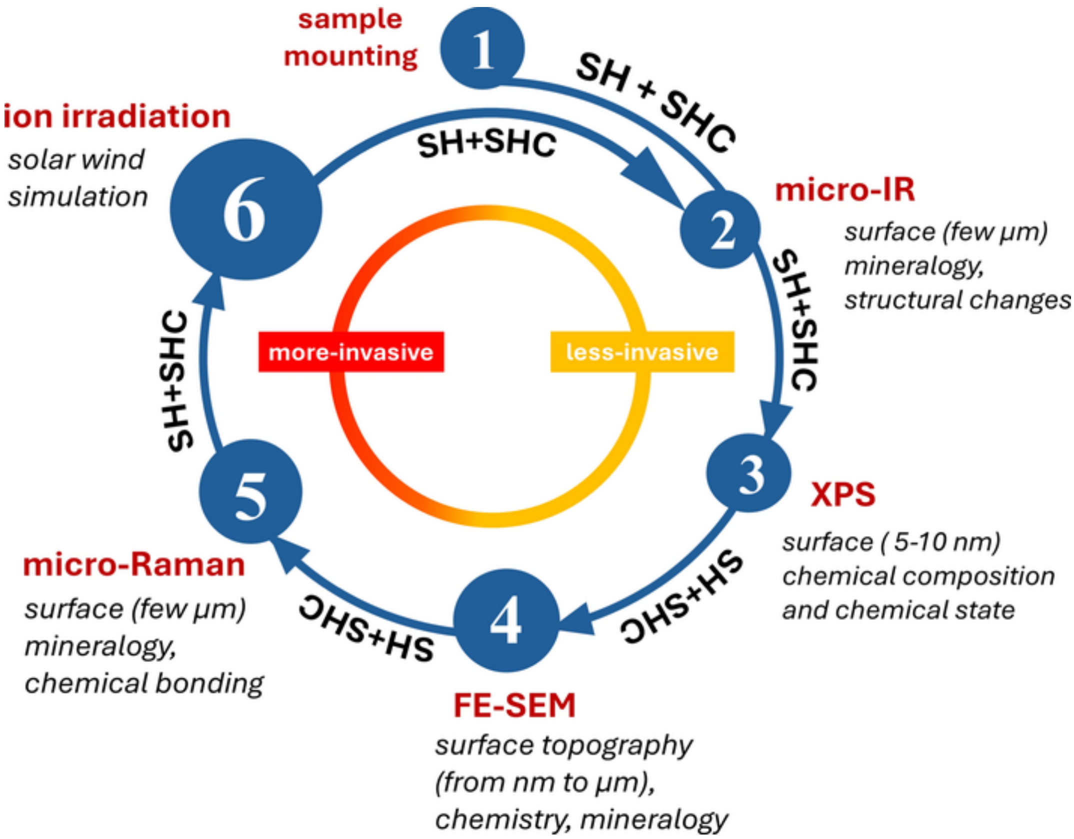
5. Development of an analytical pipeline

One of the primary objectives of this study is to apply multiple surface and sub-surface analytical techniques to a single grain while establishing a precise analytical pipeline. This approach aims to minimize artifacts introduced by the different instruments during sequential analyses. A carefully designed analytical workflow has been developed to mitigate the risk of altering the sample's physical and chemical properties. This pipeline prioritizes the use of less invasive techniques before progressing to the more invasive ones.

The sequence of analysis began with micro-IR spectroscopy, which is among the least invasive analytical techniques available to date (e.g., Rosi et al. 2019; Beć et al. 2020). Micro-IR spectroscopy generally does not induce significant physical or chemical changes and thus is particularly suitable for the preliminary investigation of returned samples (e.g., Yada et al. 2022). The XPS was employed as the second step in the analytical sequence. Indeed, XPS uses a soft X-ray source that minimizes the mobility of volatile elements, thereby reducing the risk of artifacts and ensuring more accurate results. In contrast, other analytical techniques, such as electron microscopies (e.g., FE-SEM–EDS), present limitations due to the potential mobilization of volatile elements like sodium (e.g., Goldstein et al. 2017). As highlighted by Keller and McKay (1993), the loss of these elements during FE-SEM–EDS analyses can introduce artifacts, leading to inaccuracies in the chemical data. Thus, the workflow proceeded with FE-SEM analysis, followed by micro-Raman spectroscopy. This latter employs a monochromatic laser source, causing possible localized heating that can potentially damage the sample (e.g., Smith and Dent 2019). For instance, magnetite is known to transform into hematite when exposed to a high-power laser source (Shebanova and Lazor 2003). To mitigate similar phenomena, micro-Raman analyses must be conducted at a reduced power setting (generally ≤ 1 mW, e.g., Davidson et al. 2012; Steele et al. 2007; Chan et al. 2019) and only after completing the XPS and SEM investigations to ensure minimal perturbation to the sample surface. The analytical pipeline concluded with ion irradiation (e.g., H^+, Ar^{2+}) to replicate the effects of space weathering processes. This technique simulates the impact of interplanetary solar wind, which can significantly modify the samples' surface properties, overprinting the initial workflow state (Laczniak et al. 2021). The analytical pipeline is illustrated in Fig. 1 and can be repeated in cycles, utilizing different solar wind implantation energy levels. In summary, the pipeline preserves the samples by starting with less invasive analytical techniques and gradually proceeding to the more invasive ones. This approach minimizes the risk of physical or chemical changes, ensuring accurate surface characterization and reliable results while preserving the sample's integrity.

Fig. 1

Analytical workflow specifically designed for the study of space weathering processes

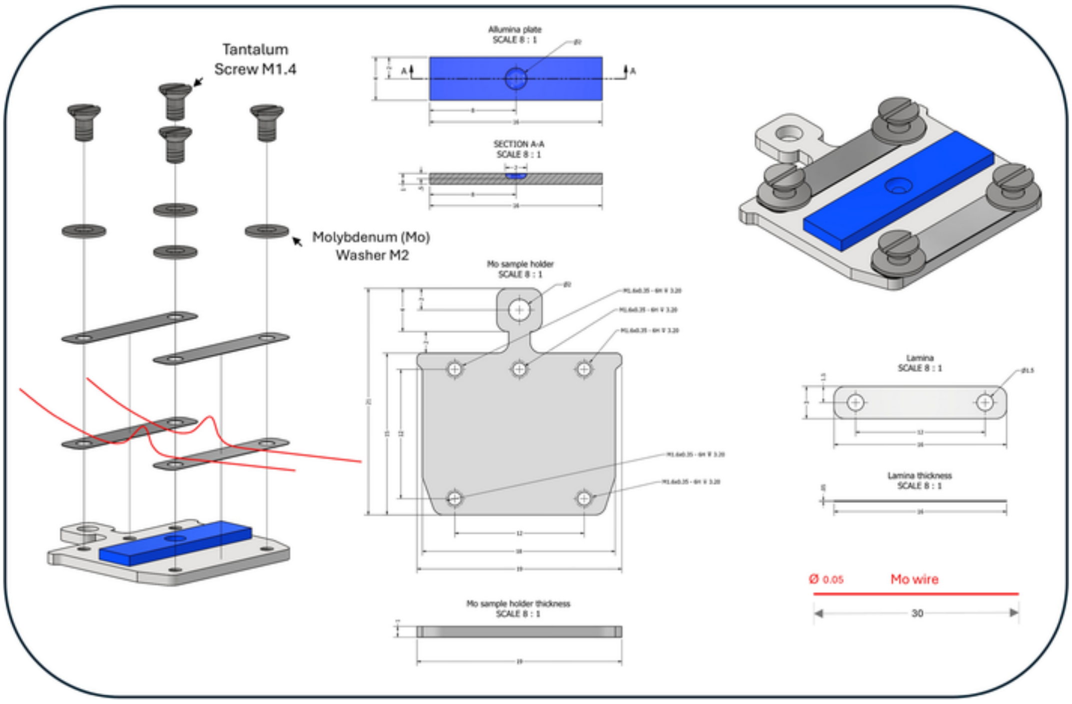


6. Design of a new SH and mounting procedures of extraterrestrial materials for the coordinated surface analyses

We developed a new SH to enhance coordinated surface analyses of extraterrestrial materials in the analytical pipeline previously presented. The SH is constructed using a molybdenum (Mo) flag-style plate (Mo-FSP), which serves as a standard holder for XPS and is also compatible with FE-SEM, micro-IR, micro-Raman, and ion irradiation. Exploded view and technical drawings of the SH are illustrated in Fig. 2. The SH assembly includes the following components: (i) Mo washers, essential for securing connections; (ii) Mo wire/s, thin Mo wire (50 µm in diameter) to facilitate fine adjustments and stability while reducing the shadow effects; (iii) Mo laminas, thin Mo sheets designed to provide structural support for the wire/s. (iv) Tantalum screws (Ta-s1 and Ta-s2), high-precision screws made of Ta to ensure robust assembly; (v) sintered alumina plate (Al_2O_3), a specifically designed plate featuring small cavities (1–2 mm in diameter) to accommodate individual grains throughout the analytical procedures safely and ensuring stable conditions during facility-to-facility transfer. Additionally, the Al_2O_3 plate can be securely attached to the top surface of the Mo-FSP using small sections of double-sided carbon tape.

Fig. 2

Exploded view and technical drawings of the sample holder components in a safety-belt-like configuration. The dimensions are provided in millimeters



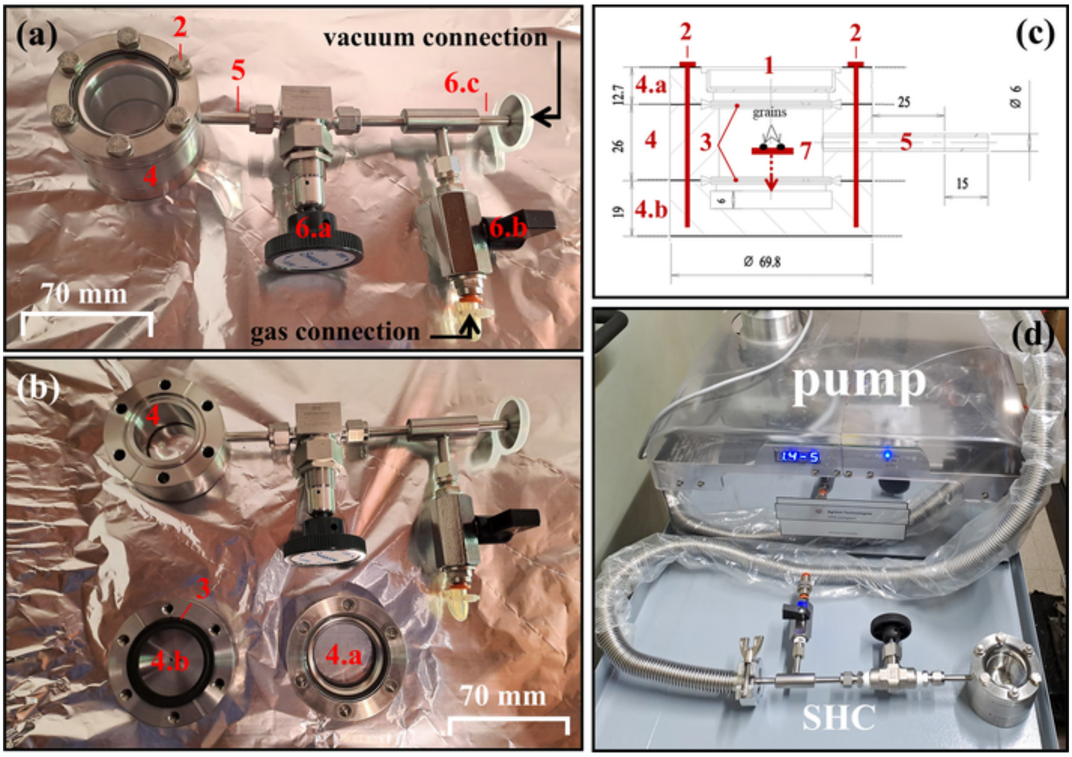
The SH design integrates these components into a versatile, modular system that ensures sample stability and protection while improving compatibility among diverse analytical methodologies. Moreover, high-pure (99.9%) Mo wire and alumina plate provide significant advantages, including high hardness, mechanical stability, and minimal degassing under UHV conditions.

7. Design of a new SHC for SH transportation and storage

The SHC was specifically designed to securely host the SH while maintaining the sample in a controlled environment to minimize terrestrial contamination. The technical drawings and photographs are presented in Fig. 3. The SHC supports two distinct environmental modes: (i) high vacuum (HV; up to 10^{-4} to 10^{-5} Pa) to prevent terrestrial moisture and atmospheric contamination, or (ii) inert gas atmosphere (e.g., N_2) to avoid oxygen-related contamination. Both environments are developed to properly preserve the samples' pristine chemical and physical properties. The SHC modular stainless steel (SS 316L) construction, comprising a main chamber, a top window (DN40CF Standard kodial glass), and a bottom chamber, ensures high vacuum compatibility and chemical resistance (Fig. 3a, b, c). The SHC is assembled using stainless-steel screws for mechanical stability, and the SH is securely fixed by a screw at the base of the bottom chamber to prevent movement during transportation among facilities (Fig. 3c). A lateral tube equipped with a high-vacuum valve, a gas valve, and connections for vacuum pumping or gas inlet, provides versatility, allowing the SHC to switch between vacuum and inert gas environments based on analytical requirements (Fig. 3d). The SHC can be assembled and sealed within a glove box filled with an inert atmosphere to prevent contamination during handling, with the high-vacuum valve maintaining the glove box environment upon sealing. Additionally, the SHC can achieve high-vacuum conditions not only for storing the samples, but also for cleaning the chamber and refilling it with inert gas (e.g., N_2 or Ar). This flexible design ensures the SHC maintains sample integrity during storage and inter-laboratory transport.

Fig. 3

Photographs and technical drawing of the SHC. **a** Photograph of the assembled SHC. **b** Photograph of the main components of the SHC before the assemblage. **c** Technical drawing of the SHC. The lengths (black color numbers) are expressed in millimeters. The red color numbers indicate the different SHC components, described as follows: 1. glass window; 2. stainless-steel screws; 3. Viton rubber; 4. stainless-steel chamber of the SHC; 4a. top window; 4b. bottom chamber; 5. stainless tube; 6a. high-vacuum valve; 6b. gas connection and gas valve; 6c. vacuum pumping system connection; 7. sample holder fixed by a stainless-steel screw at the base of the bottom chamber. **d** photograph of the SHC connected to the vacuum turbo pumping system



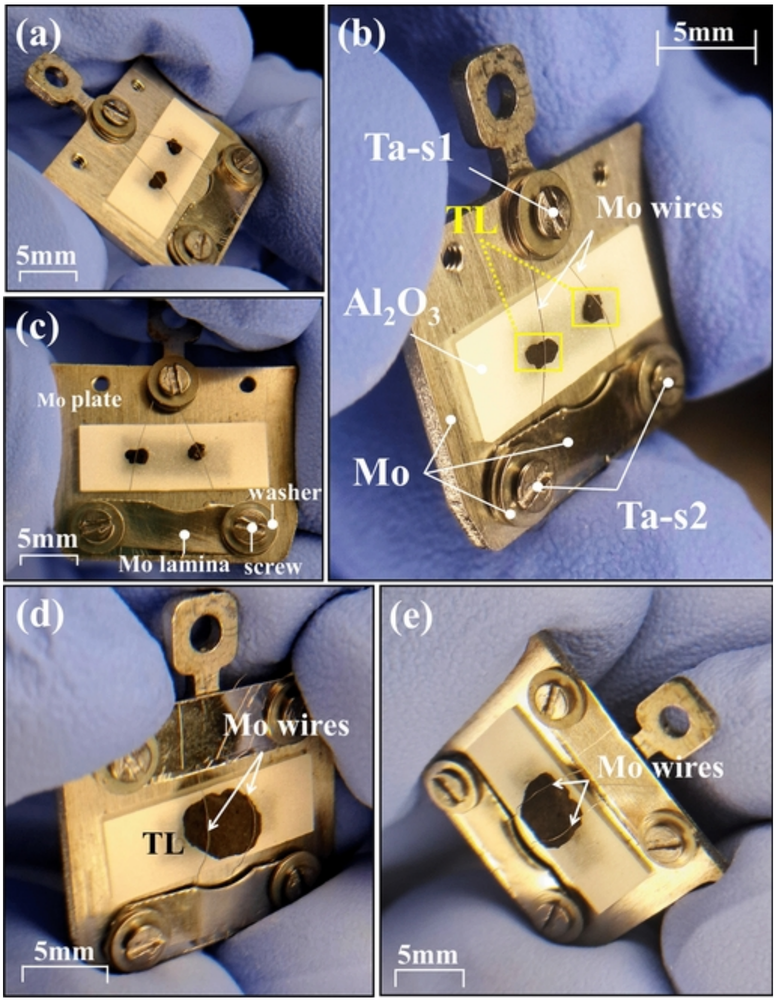
8. Mounting procedure and SH testing

To evaluate the performance of the SH, mounting tests were performed using Tagish Lake meteorite grains, chosen for their similarity to Ryugu and Bennu grains in density and friability (Blinova et al. 2014; Yada et al. 2022; Yokoyama et al. 2023; Lauretta et al. 2024).

The grain is mechanically secured between the Al_2O_3 plate and Mo wire/s (Fig. 4). A single Mo wire is used for grains smaller than 2.5 mm (Fig. 4a, b, c), while two Mo wires are recommended for grains larger than 4 mm (Fig. 4d, e). For intermediate sizes, the choice between single-wire or double Mo wire configurations (safety-belt-like) depends on the geometry of the grain. The illustrated setup eliminates the need for additional constraints or tools except for a precision metal tweezer with fine-cut tips (TFCT) and specific screwdriver for tightening Ta screws to secure the wire in position.

Fig. 4

Photographs of the SH configuration for XPS and other surface analytical techniques. **a**, **b**, **c** Configuration for < 2.5 mm in size. The Tagish Lake grains are fixed between the Al_2O_3 plate and the molybdenum (Mo) wire (50 µm in diameter). Tantalum-s1 (Ta-s1) is the pin screw, while Ta-s2 represents the movable screws. When the grain-wire configuration is steady, the Ta-s2 screws are definitively fixed. **d**, **e** Grains larger than 4 mm can be mounted using two Mo wires arranged in a safety-belt-like configuration



For grains smaller than 2.5 mm, Ta screw n. 1 (Ta-s1) functions as a pin to anchor the wire, while the Ta screws n. 2 (Ta-s2) pass through two Mo laminas and provide adjustable tension (Fig. 4b). The Mo wire remains movable and can be positioned freely until Ta-s2 is tightened, thus securing the wire-grain configuration.

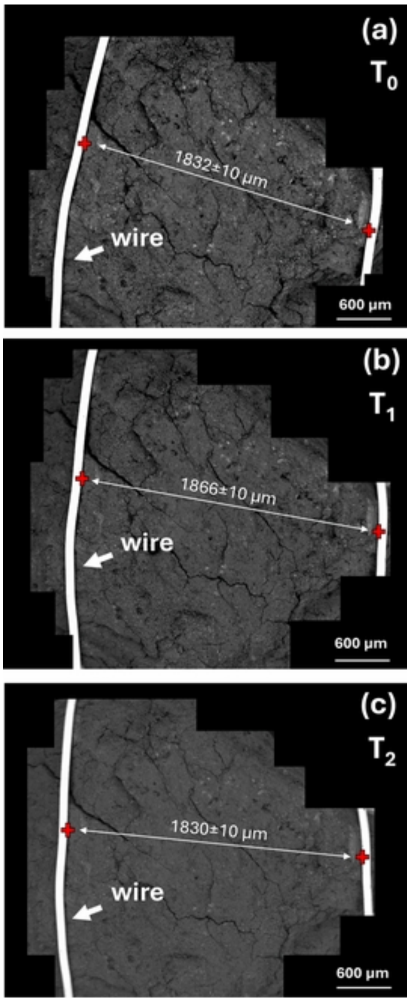
The grain is first placed into the Al_2O_3 cavity. The Mo wire is then carefully oriented above the grain, and its end is gently pulled using TFCT until the Mo wire touches the surface of the grain. Once in position, Ta-s2 is tightened to secure the configuration (Fig. 4a, b, c). This configuration also allows the potential mounting of two grains in a single SH for grains smaller than 2.5 mm, as shown in Fig. 4b, optimizing the coordinate analysis. For grains larger than 4 mm, a double Ta-s2 configuration is recommended (Fig. 4d, e). The latter ensures enhanced stability and better distribution of mechanical stress.

9. Performance and stability of the SH

When grains are correctly mounted, the SH ensures prolonged stability during extended and coordinated investigations and laboratory simulations (e.g., ion bombardment). We evaluated the drifting motion over three consecutive cycles of coordinate analysis using the previously illustrated analytical workflow (Fig. 1). This pipeline, which includes micro-IR, XPS, FE-SEM, micro-Raman, and ion irradiation techniques, was performed at progressively increasing irradiation fluences. As shown in Fig. 5, no significant drift was observed after irradiation at 400 keV Ar^{2+} with a fluence of $1 \cdot 10^{15}$ ions/cm^2 and after at 400 keV Ar^{2+} with a fluence of $3 \cdot 10^{15}$ ions/cm^2. The grains exhibited minimal displacement, confined to a range of ~ 10 microns. This result shows the robustness of the SH under diverse irradiation conditions, confirming its suitability for measurements in different environments: UHV through XPS analysis, ions irradiation during solar wind simulation, HV for FE-SEM, and atmospheres pressure for micro-IR and micro-Raman.

Fig. 5

Backscattered electron images acquired during coordinated analyses and solar wind simulation experiments with increasing fluence on a Tagish Lake meteorite grain. The sample was mounted in the safety-belt-like sample holder configuration, as shown in Fig. 4d. **a** T_0 represents the unirradiated state at time zero. **b** T_1 corresponds after the first irradiation at 400 keV Ar^{2+} with a fluence of $1 \cdot 10^{15}$ ions/cm^2. **c** T_2 corresponds after the second irradiation at 400 keV Ar^{2+} with a fluence of $3 \cdot 10^{15}$ ions/cm^2. The red cross marks the reference points used to measure the distance between wires after completing the workflow cycle shown in Fig. 1. Errors are expressed as 1σ, and the uncertainties correspond to the last significant figures



10. The versatility and practicality of the SH

The SH is designed to offer high versatility and practicality. It can be assembled straightforwardly, even within a glove box, under a controlled atmosphere. The assembly process is entirely mechanical, requiring only a screwdriver and precision tweezer, making it accessible and user-friendly. The SH was initially designed to be compatible with a universal holder for XPS analysis but is easily adaptable to other analytical techniques. All components are readily available from specialized suppliers of XPS or UHV equipment.

Mounting samples is relatively simple for grains larger than 4 mm in size; however, it becomes more challenging for smaller grains (< 2.5 mm). For grains of intermediate sizes, the choice of the mounting configuration (e.g., single-wire or safety-belt-like) depends on the geometry of the grain. For instance, a safety-belt-like configuration may be preferable for elongated grains.

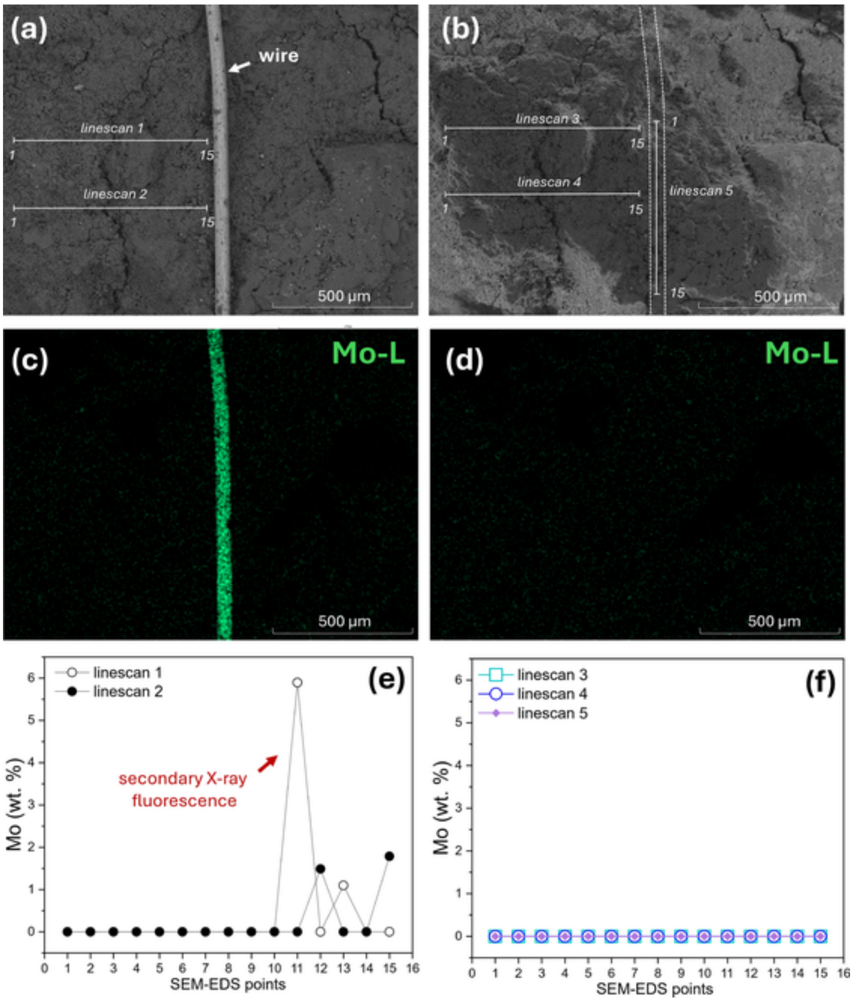
Due to carbonaceous materials' highly friable nature, improper assembly handling can compromise the sample integrity. Therefore, we warmly recommend practicing the mounting procedure using analogous specimens before proceeding to the extraterrestrial samples. As illustrated above, the SH can accommodate grains of various sizes (e.g., 1.5–10 mm) and geometries, offering great versatility. This, combined with the SH's robust and easily implementable design, makes it a valuable tool for multidisciplinary research requiring precise handling and stability under different experimental conditions.

11. Evaluation of contamination induced by the SH

We utilized high-purity (99.9%) alumina (Al_2O_3) plate and Mo wire to mitigate the risk of chemical contamination by the SH components. High-hardness materials, such as sapphire (Al_2O_3), were chosen as the optimal holder for transporting Ryugu grains between facilities (Okazaki et al. 2022). Additionally, to ensure that the Mo wire used in the SH did not compromise the integrity of the grains, we investigated potential contamination by performing SEM-EDS analysis (Fig. 6a, b). We specifically mapped the regions where the wire interfaced with the grain, and the results revealed no detectable traces of Mo particles (below detection limit = 0.1 wt. %; Fig. 6c, d). This suggests the wire did not introduce significant or measurable contamination to the sample surface. However, secondary X-ray fluorescence from Mo was observed in the wire's nearness (up to 400 µm). This effect can be attributed to the irregular topography of the unprepared samples (Goldstein et al. 2017), which likely led to significant secondary X-ray fluorescence emission by Mo wire, as shown in Fig. 6e.

Fig. 6

Electron backscattered images acquired with (**a**) and without (**b**) the Mo wire. **c**, **d** Corresponding SEM–EDS maps showing the Mo Lline (detection limit of 0.1 wt. %) for images **a** and **b**, respectively. The EDS maps were processed using the "TruMap" function of the AZtec software. **e**, **f** SEM–EDS linescans plotting Mo (wt. %) along the highlighted sections in images **a** and **b**



12. Evaluation of the shadow effects induced by the SH

The presence of the Mo wire on the sample surface can introduce shadowing effects, potentially leading to analytical artifacts. This section evaluates the impact of shadowing effects, focusing on the areas where these effects are more pronounced due to the wire crossing the sample surface. In contrast, the SH safety-belt-like configuration does not produce noticeable shadowing effects, as the central surface remains fully exposed for coordinated analyses (i.e., Fig. 4e).

12.1. XPS analysis

High-resolution Fe 2p photoelectron spectra were acquired using grains of the Tagish Lake meteorite, with sizes < 2.5 mm and > 4 mm (Fig. 4). This analysis aimed to evaluate potential shadowing effects, as the chemical state of iron serves as a key indicator for assessing space weathering effects (e.g., Laczniak et al. 2021, 2024). The Fe 2p spectra for both grain sizes exhibit characteristic peaks corresponding to the spin–orbit coupling (Fe $2p_{3/2}$ and Fe $2p_{1/2}$) (Fig. 7), confirming that the SH does not introduce analytical artifacts into the XPS spectrum. To evaluate the potential shadowing effects of the SH's Mo wire, the Mo photoelectron contribution was monitored for the grain < 2.5 mm. Weak Mo 3d peaks were detected (Fig. 8), but they were negligible due to the small area of the wire compared to the grain surface. Moreover, the Mo 3d peaks do not overlap with key elements of interest, such as Fe 2p, S 2p, Na 1 s, and Si 2p (Chastain and King 1992), ensuring they do not interfere with the spectral analysis.

Fig. 7

Iron 2p photoelectron spectra of the < 2.5 mm (blue) and > 4 mm (black) Tagish Lake grains

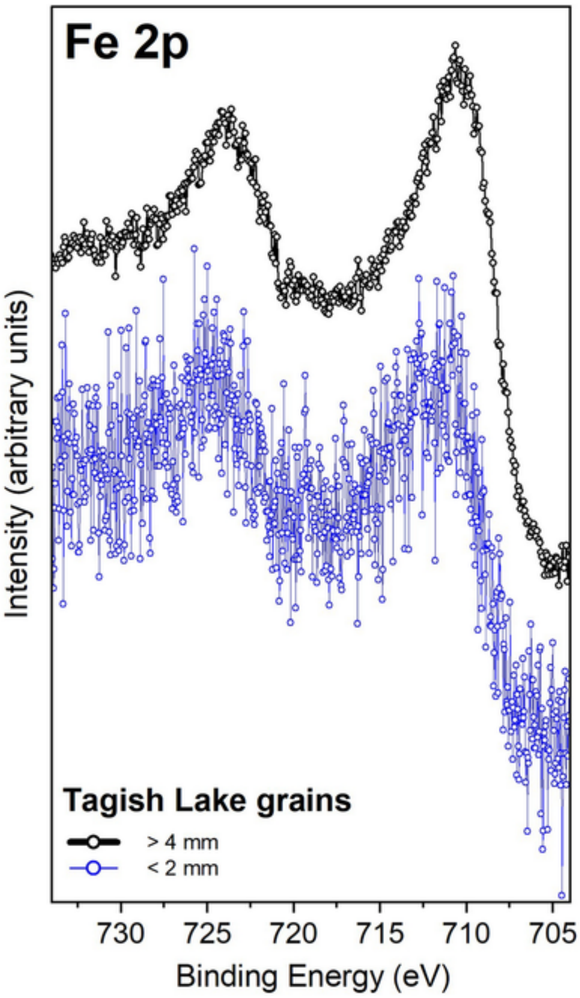
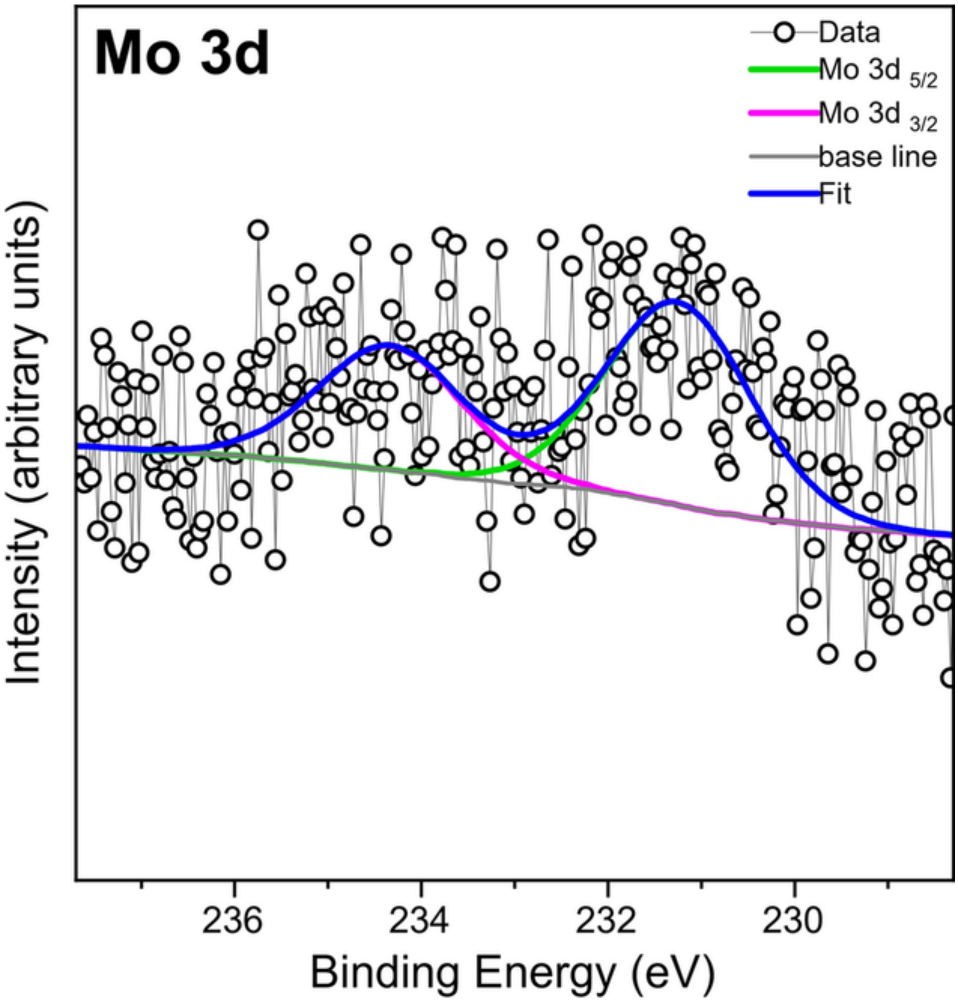


Fig. 8

Molybdenum 3d photoelectron spectra of the Tagish Lake grain (< 2.5 mm in size)



12.2. Micro-IR and micro-Raman spectroscopy

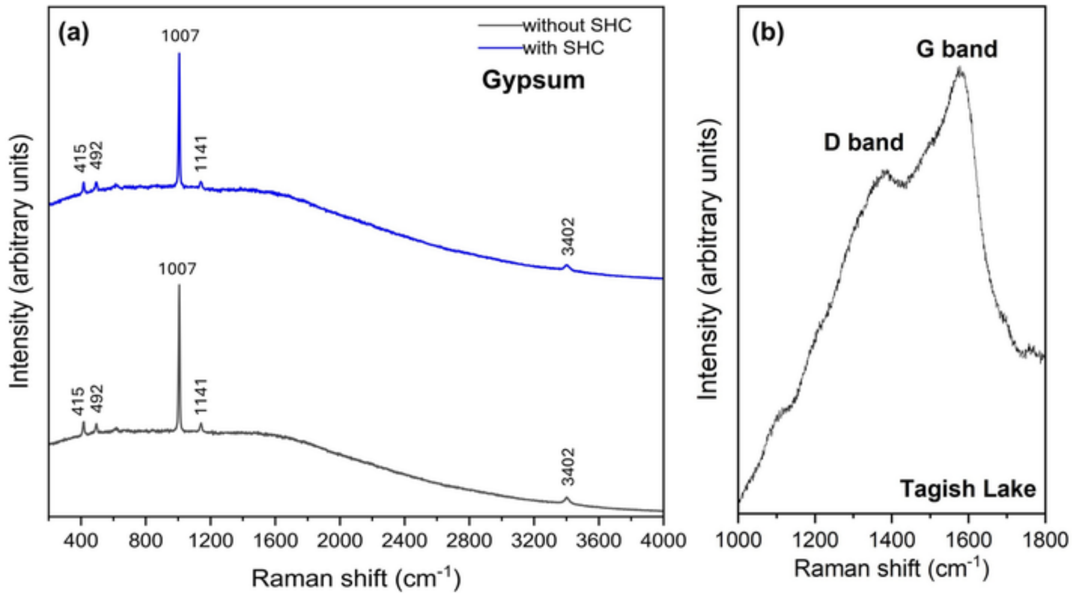
Micro-IR and micro-Raman spectroscopy techniques typically employ radiation oriented perpendicularly to the sample surface, thus limiting shadowing effects to the region near the wire. During spectroscopic measurements, external objects like the Mo wire can cast shadows depending on the angle of signal incidence. These shadowing effects are analogous to natural phenomena observed on a larger scale, such as shadows cast by surface boulders on asteroids (e.g., Sugimoto 2021). Additionally, intrinsic sample properties—such as high porosity and surface roughness—can cause deviations from the ideal signal behavior, as described by Hapke (1981, 2008). These factors can make it challenging to isolate individual effects on the signal.

To mitigate shadowing effects, the SH used a wire of minimal thickness. This ensures its impact on the signal remains negligible while securely holding the sample.

Additionally, micro-Raman analysis offers the distinct advantage to perform measurements directly within the SHC, offering an inert environment, enabling precise data collection minimizing external contaminants. This capability is given by the SHC window, which is made of synthetic glass transparent to the laser wavelength (~ 632 nm), typically used in micro-Raman analyses. As shown in Fig. 9a, the gypsum spectra obtained with and without the SCH are indistinguishable. This result confirms that the SHC glass window does not introduce spectral artifacts. This is further supported by Raman measurements conducted on the SHC using the Tagish Lake meteorite, as evidenced by the characteristic D and G bands of the carbon compounds, as shown in Fig. 7b and reported by Chan et al. (2019).

Fig. 9

Raman spectra of gypsum acquired with and without SHC (**a**) and of the Tagish Lake grain on the SHC (**b**)



12.3. FE-SEM

As previously mentioned, the shadowing effects from the SH are primarily localized near the Mo wire. This is not a significant limitation, as FE-SEM investigations typically target up to nanoscale regions, particularly during imaging analyses. Nevertheless, the irregular topography of unprepared samples can lead to secondary X-ray fluorescence (SXF) phenomena when the analyzed area is close (up to 400 µm) to the Mo wire or near the alumina plate, potentially introducing artifacts in EDS analysis, as shown in Fig. 6. However, this effect can be mitigated by adequately adjusting the sample-detector geometry by SEM specimen stage. By optimizing this configuration, the impact of SXF can be minimized, ensuring more accurate EDS results. Additionally, the presence of electrically conductive material, such as Mo wire, minimizes charging effects on the sample's surface, ensuring accurate SEM investigations, as shown in Fig. 6a,b.

12.4. Ion irradiation

The SH was aligned orthogonally to the ion beam, minimizing shadowing effects on the grain and enhancing the uniformity of ion irradiation. Additionally, the ion current was carefully controlled, remaining below 400 nA/cm^2, to prevent any surface heating of the grain.

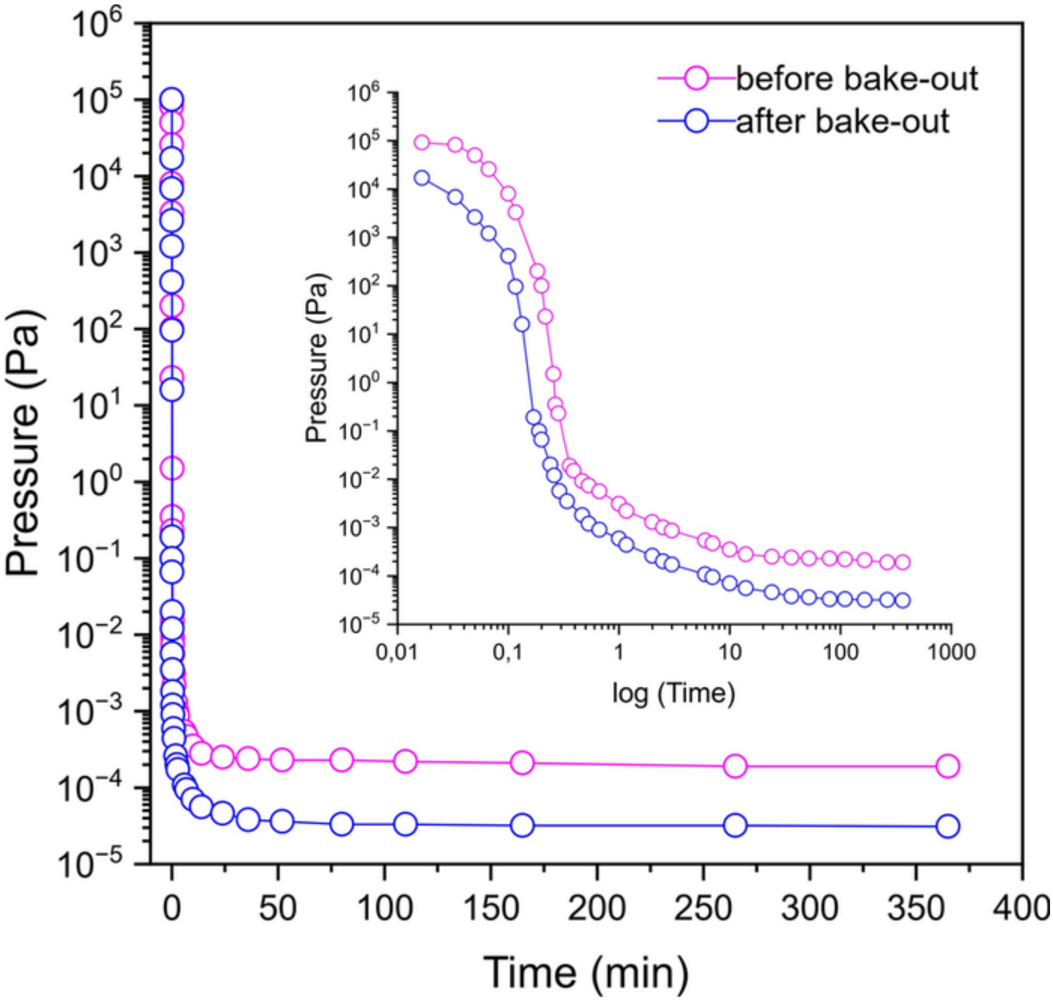
13. Performance and testing of the SHC

The SHC was tested to evaluate its performance in preserving sensitive samples, such as extraterrestrial materials, under various environmental conditions.

Dynamic vacuum tests conducted before and after a 24-h bake-out at 373.15 K demonstrate the SHC's capacity to achieve high-vacuum conditions (from 10^{-4} to 10^{-5} Pa), with the bake-out significantly improving vacuum quality by reducing outgassing from stainless-steel components (Fig. 10).

Fig. 10

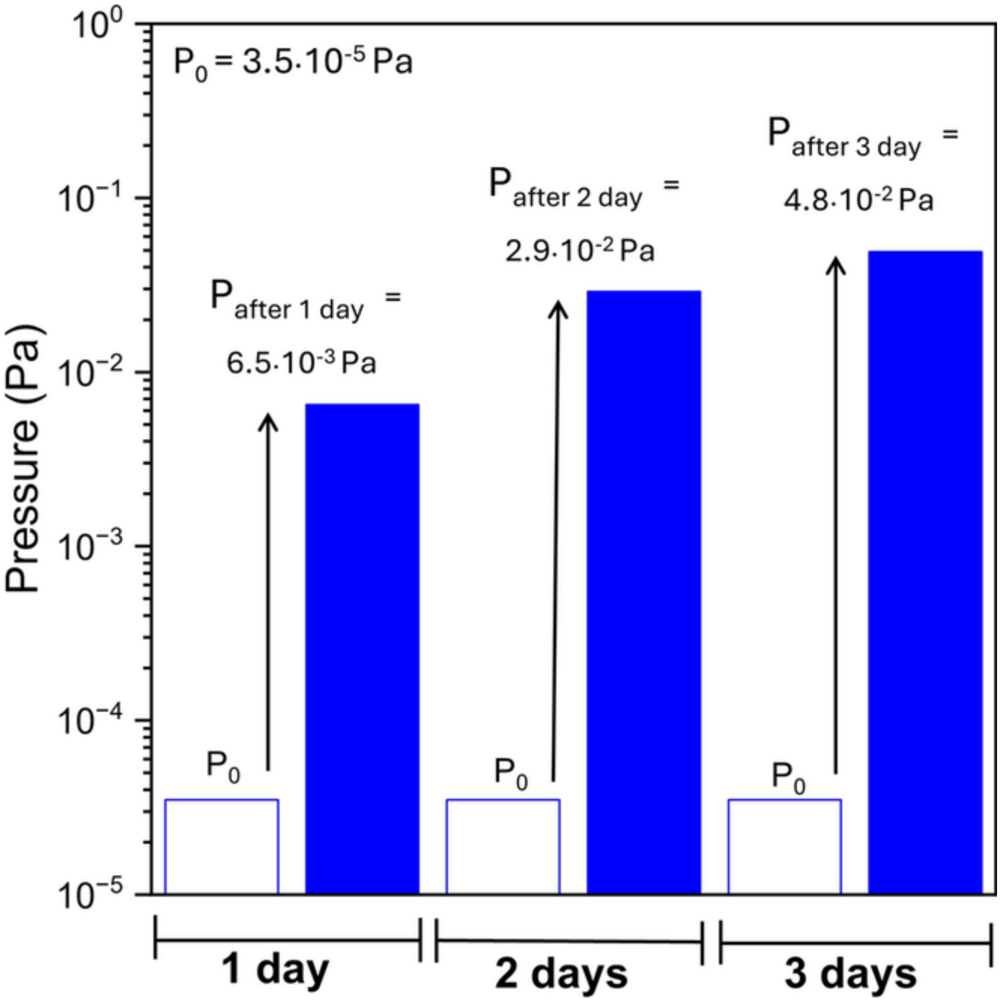
Pressure versus time semi-log and log–log graphs. The plots were obtained during the pump-down of the SHC before and after the bake-out procedure



Static vacuum tests simulated real scenarios, such as transport without active pumping, revealing a predictable pressure increase due to hydrogen outgassing, possibly released by the SHC components (e.g., Redhead 1999). As illustrated in the pressure-versus-time diagram in Fig. 11, under static conditions, the pressure of the SHC increased from $3.5 \cdot 10^{-5}$ to $6.5 \cdot 10^{-3}$ Pa after 1 day, from $3.5 \cdot 10^{-5}$ to $2.9 \cdot 10^{-2}$ Pa after 2 days, and from $3.5 \cdot 10^{-5}$ to $4.8 \cdot 10^{-2}$ Pa after 3 days.

Fig. 11

Graph showing the SHC sealing pressure test under high vacuum static conditions (i.e., without using the pumping system). The white columns represent the test starting points under ~ $3.5 \cdot 10^{-5}$ Pa pressure (P_0), and the blue columns show the test final points for the different periods ($P_{\text{after n. day/s}}$)



Based on these findings, we suggest storing samples in dynamic vacuum conditions within the SHC at a pressure of ~ 10^{-5} Pa after the analyses. Additionally, we recommend achieving the vacuum condition through a gradual pump-down to minimize the turbulence effects and ensure the stability of the grain mounting.

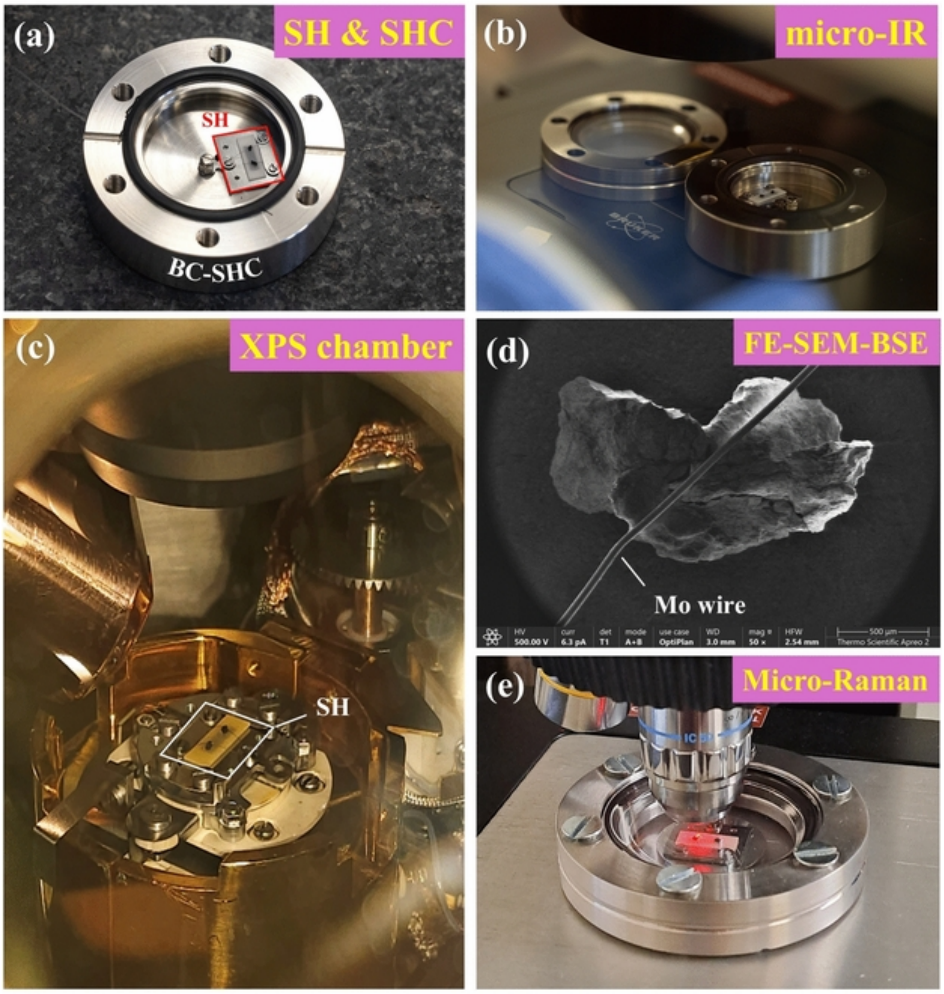
During transportation between facilities, the SHC can maintain samples under static vacuum conditions, with pressure varying depending on the duration of transport (Fig. 11). However, the SHC is also designed to preserve samples under inert gas atmospheres at atmospheric pressure, providing a practical storage environment to prevent terrestrial contamination. These capabilities make the SHC highly versatile for securely preserving and transporting sensitive samples.

14. Coordinated analyses of Ryugu grains

Two Ryugu grains, collected during two different touchdowns by the Hayabusa2 spacecraft, were analyzed using coordinated surface analyses to study the effects of space weathering. The grains, catalogued by JAXA as A0226 and C0242, were transferred from their original container (Ito et al. 2020) and mounted on the SH under an N_2 atmosphere within a glove box to preserve their pristine state (Palomba et al. 2024). We mounted the two grains in a single SH plate to optimize the analytical sequences using the configuration presented in Fig. 12. The previously described pipeline was adopted to ensure less analytical damage on the sample. Sample transfers between facilities were conducted using the SHC under an inert N_2 or Ar atmosphere to minimize exposure to terrestrial contamination. Additionally, the samples were transferred from the SHC to the analytical instruments within a glove box or using specialized sample-transfer systems. This sequence preserves the samples' pristine characteristics throughout the different analytical measurements.

Fig. 12

Photographs of the Ryugu grains during the coordinate analyses mounted in the SH described in this study (Fig. 4b). **a** The SH containing the Ryugu grains fixed at the bottom chamber of the sample holder container (BC-SHC). Photographs under measurements: micro-IR analysis (**b**), XPS analysis (**c**), FE-SEM analysis; electron image microscope of A0226 Ryugu grain crossed by the molybdenum wire of the SH (**d**), micro-Raman (**e**)



15. Summary and final remarks

This study presents the development and implementation of a new SH and SHC specifically designed to enhance the coordinated surface analyses of extraterrestrial materials while maintaining samples' integrity. The SH enables secure mounting of irregular and fragile grains for sequential analyses across various advanced techniques, including micro-IR, XPS, FE-SEM, micro-Raman spectroscopy, and ion irradiation. Its modular design incorporates high-purity materials, such as Mo and alumina (Al_2O_3), ensuring minimal contamination and high stability under UHV and inert gas environments. The SHC complements the SH by offering a robust and adaptable solution for inert-laboratory transport, capable of maintaining HV or inert gas conditions to prevent terrestrial contamination.

This work also presents an analytical pipeline carefully designed to minimize the risk of physical or chemical alterations to the sample during sequential analyses. This workflow prioritizes less invasive techniques, such as micro-IR, followed by XPS, FE-SEM, micro-Raman, and progressing to the more invasive ion irradiation processing as the final step.

Performance tests using analog materials and Tagish Lake grains demonstrated the SH's capability to maintain grain stability with minimal drift and no significant contamination or shadowing effects. Furthermore, the SHC effectively preserved sample integrity under vacuum and inert gas conditions, ensuring safe transport and storage.

Finally, the SH and SHC were applied to the coordinated surface analysis of Ryugu grains, successfully preserving their integrity while facilitating a comprehensive examination of the space weathering effects. These innovations, combined with the analytical pipeline, provide a reliable framework for multidisciplinary research on sensitive extraterrestrial materials, ensuring accurate and precise results and the integrity of these precious samples.

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Author contributions

X. S. conceptualization, formal analysis, data curation, investigation, methodology, visualization, roles/writing—original draft. G. P. conceptualization, methodology, resources, supervision, roles/writing—original draft. S. C. conceptualization, formal analysis, methodology, writing—review and editing. B. C. investigation, methodology. S. R. investigation, methodology, writing—review and editing. C. S. investigation, methodology. G. B. formal analysis, investigation, resources, writing—review and editing. M. A. investigation, methodology, writing—review and editing. F. D: writing—review and editing. A. S. formal analysis, investigation. M. R. investigation. G. V. investigation. G. O. investigation. A. L. writing—review and editing. T. C. formal analysis, investigation. A. F. writing—review and editing. E. P. funding acquisition, methodology, resources, writing—review and editing.

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Data availability

The data are contained within the manuscript.

Declarations

Ethics approval and consent to participate

Not applicable.

Consent for publication

Not applicable.

Competing interests

The authors declare no competing interests.

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