


TECHNICAL REPORT

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A curation for uncontaminated Hayabusa2-returned samples in the extraterrestrial curation center of JAXA: from the beginning to present day

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Abstract

Developing a cleanroom and clean chambers (CCs) for Hayabusa2 returned samples has been discussed with the committee for Hayabusa2 sample curation facility since 2015. One major difference from the specifications of the CCs used for Itokawa samples is that a part of samples was decided to be handled and preserved in vacuum to avoid terrestrial nitrogen contamination with organics or unknown materials that might easily react with the samples. Thus, the CCs for Hayabusa2 samples were divided into two CCs for vacuum processes and three CCs for purified nitrogen conditions. The cleanroom was built in summer 2017, while the CCs were installed in the summer of 2018. After the installation of the CCs, instruments for initial descriptions, sample containers, handling tools for powder and particle samples, and jigs to assist handling samples were developed in parallel with functional checks and repeated rehearsals between the fall of 2018 and the fall of 2020. The curatorial works on Hayabusa2-returned samples were conducted as previously planned. Simultaneously, contaminations and influences of inorganics, organics, microbial, and magnetic constructs have been assessed to evaluate their potential effects on the analysis of the returned samples. Additionally, the tools used to touch samples directly have been demagnetized to avoid sample magnetization during their handling and the tool magnetization was measured before and after their usages. The series of developments and experiences from the curatorial works of Hayabusa2-returned samples represent valuable implications for future sample return missions.

Keywords Hayabusa2, Ryugu, C-type asteroid, Curation, Cleanroom, Clean chamber

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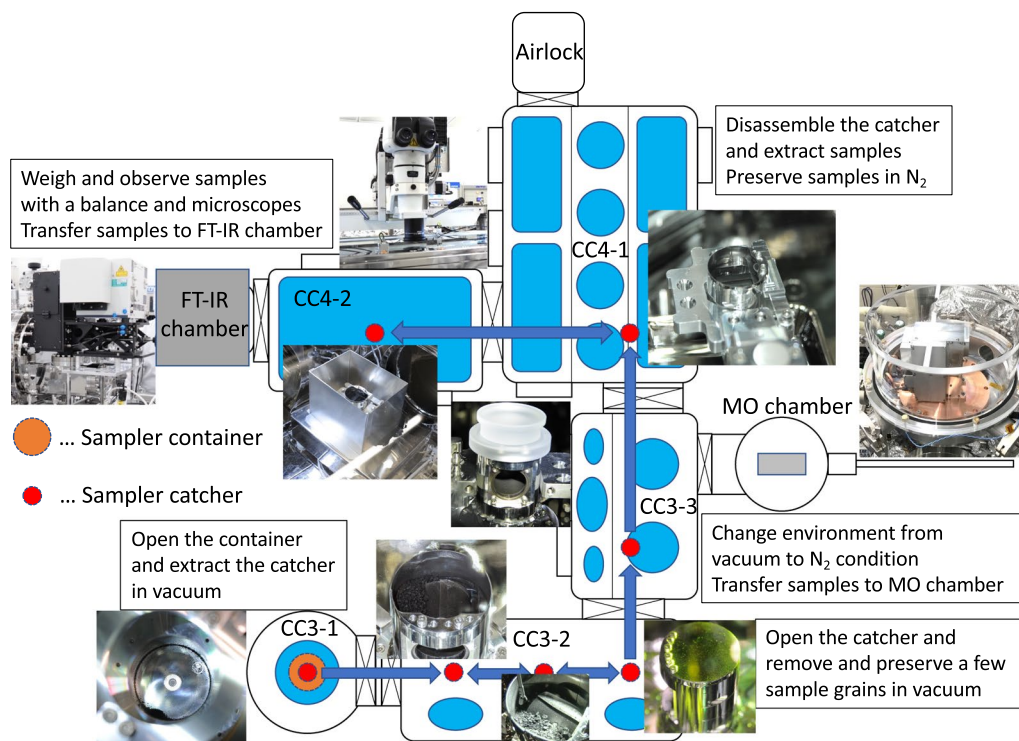
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Graphical Abstract



Introduction—requirement for handling Hayabusa2-returned samples

The first Hayabusa spacecraft (HY1) accomplished a sample return from the S-type near-Earth asteroid Itokawa back to Earth on June 13, 2010 (Yada et al. 2014). Initial analyses of the Itokawa samples indicated that they were comparable to equilibrated LL chondrites (Nakamura et al. 2011; Yurimoto et al. 2011; Ebihara et al. 2011), revealing features of space weathering including solar wind implantation and micrometeoroid impacts (Noguchi et al. 2011; Nakamura et al. 2012; Nagao et al. 2011) as well as regolith gardening processes (Tsuchiyama et al. 2011). More than 1000 individual Itokawa particles have been collected and described (Yada et al. 2014; 2021). Among them, more than 200 particles have been distributed to principal investigators of international announcement of opportunity (AO), which led to significant scientific discoveries in a wide range of fields (i.e., Langenhorst et al. 2014; Thompson et al. 2014; Keller and Berger 2014; Mikouchi et al. 2014; Bonal et al. 2015; Park et al. 2015; Fujiya et al. 2016; Jourdan et al. 2017; Böttger et al. 2017; Terada et al. 2018; Matsumoto et al. 2018; Daly et al. 2021). This includes the first discovery of water in an S-type asteroid (Jin and Bose 2019)

and possible existence of indigenous organics (Chan et al. 2020), which ultimately help us to better understand our solar system. These scientific results are based on the preservation and handling of asteroidal returned samples under non-atmospheric conditions. Thus, this highlights the important roles of curatorial works that contribute to the knowledge of planetary sciences.

Hayabusa 2 (HY2), which reached the near-Earth C-type asteroid 162173 Ryugu and returned asteroidal materials, was launched on December 3, 2014 (Tsuda et al. 2020). The committee for the HY2 sample curation facility was established in 2015 and was commissioned with the design of the basic concept of the handling facility intended to obtain maximum scientific outcomes from the returned samples (this committee lasted for a year). Ground-based observation of the target asteroid Ryugu indicated that its infrared spectrum was categorized to be a C-type, closely related to carbonaceous chondrites (Vilas 2008; Pinilla-Alonso et al. 2013). Carbonaceous chondrites contain up to 3.96% of organic carbon (Grady et al. 2002); thus, analyzing organics is one of the primary scientific goals of the returned sample. Therefore, the HY2 mission sampler team requested that small portions of samples should be exclusively treated under vacuum

conditions to avoid terrestrial nitrogen contamination of the extraterrestrial organics or unknown material that might be contained within samples. An HY2 sampler container can be sealed under vacuum conditions and store a sample catcher including returned samples. The sampler catcher, which would include returned samples inside, is equipped with three chambers (A, B, and C) for storing samples from different sampling sites (Sawada et al. 2017). The sampler team required the recovery of a few grains of samples from chamber A of the sampler catcher.

Alternatively, major portions of samples were planned to be treated and preserved in purified nitrogen conditions as handling samples under such conditions is easier than in vacuum and allows the prompt distribution of samples—unexposed to the terrestrial atmosphere—to the initial analysis team. To handle samples, the mission sampler team requested HY2 curation to avoid using plastic and use the least amount of polytetrafluoroethylene (PTFE) or perfluoroalkoxy alkanes (PFA) to prevent plasticizer contamination. Additionally, the team advised against electron microscopic analyses to avoid organic contamination on samples' surfaces due to electron irradiation in the low vacuum conditions of the analysis chamber of the electron microscope. The curation facility for HY2 was briefly summarized by McCubbin et al. (2019) and Yada et al. (2022a). In this report, we describe specifications and design details of the cleanroom and clean chambers (CCs), as well as the preparation steps and procedures set in place for the curation of HY2-returned samples.

Specifications of the cleanroom and clean chambers for curation: a case of Hayabusa2-returned sample

Based on the concept authorized by the consulting committee for the HY2 sample curation facility, a cleanroom for HY2-returned samples was designed to adhere to the ISO class 6 cleanliness and to be equipped with a grating floor as that used for the HY1-returned samples (Yada et al. 2014). Figure 1 illustrates the floor plan designed for the new cleanroom for HY2-returned samples. Although it was designed to be under downflow pressure from the clean room for the HY1-returned samples, the cleanroom was maintained at 15–20 Pa positive pressure compared to the normal room to prevent inward flow from the normal room, which may cause potential contamination with terrestrial detritus particles. Table 1 shows materials used for the HY2 cleanroom; all of them are composed of materials that release low amounts of gas. Except for floor painting materials, most materials are the same as those used for the HY1 sample cleanroom (Yada et al. 2014). The furniture, floors, and walls of cleanrooms have been cleaned using ultra-pure water, at least once every other

week. Based on the specification authorized by the consulting committee, the cleanroom for HY2 clean chambers has been constructed between April and November 2017 and the clean chambers have been manufactured between March 2017 and March 2018 (Table 2).

The CCs for HY2 were designed to have five different components that are connected serially (Fig. 2). Two CCs (CC3-1 and CC3-2) were designed for sample storage under vacuum condition, whereas three CCs (CC3-3, CC4-1, and CC4-2) were designed for sample processing under purified nitrogen condition. The number of CCs for HY2 samples starts from three because CC1 and CC2 were used for the HY1 samples. CC3-1 and CC3-2 were designed to open the HY2 sampler container and sampler catcher as well as extract a few particles and store them under vacuum condition. Next, the catcher containing most of the samples was planned to be transported to CC3-3 where the chamber environment changes from vacuum to purified nitrogen. Impurities such as H₂O, O₂, CO₂, and CH₄ in the nitrogen gas supplied to the CCs were less than a few parts per billion (ppb). Under purified nitrogen condition (in CC4-1), the catcher is disassembled to recover samples from each chamber. Samples are mainly handled in CC4-2 with a flat top view window, which allows easy observation of samples in the chamber using an optical microscope.

These new chambers were installed into the new cleanroom intended for the HY2 samples, which has been constructed adjacent to the cleanroom for HY1 samples (Yada et al. 2014). Materials used for instruments, including the CCs for HY2-returned samples, are listed in Table 1. We managed to decrease the number of materials used for the CCs to minimize terrestrial-origin contaminants within the returned samples. The main material used for manufacturing the CCs was stainless steel 304, whose inner surfaces was electrochemically polished (Fig. 3). All chambers were set in the cleanroom in August 2018. A new glove for the CCs of HY2 was selected based on organic solution tests, because the pure Viton gloves used for HY1 CCs went out of production (product company, Honeywell International Inc.). Alternative to pure Viton gloves, few gloves were tested using dichloromethane (DCM) solution. A total volume of 200 μ L DCM solution was dropped on the surface of a palm part of tested gloves, and 2 μ L out of the 200 μ L DCM solution was analyzed using gas chromatograph mass spectroscopy (GC–MS) (Fig. 4). Our analysis showed that only a few organic compounds, N-alkanes, were detected from the solution of a butyl glove coated with Viton produced by JUGITEC, a German glove company. Thus, this glove was considered an excellent alternative to the pure Viton gloves.

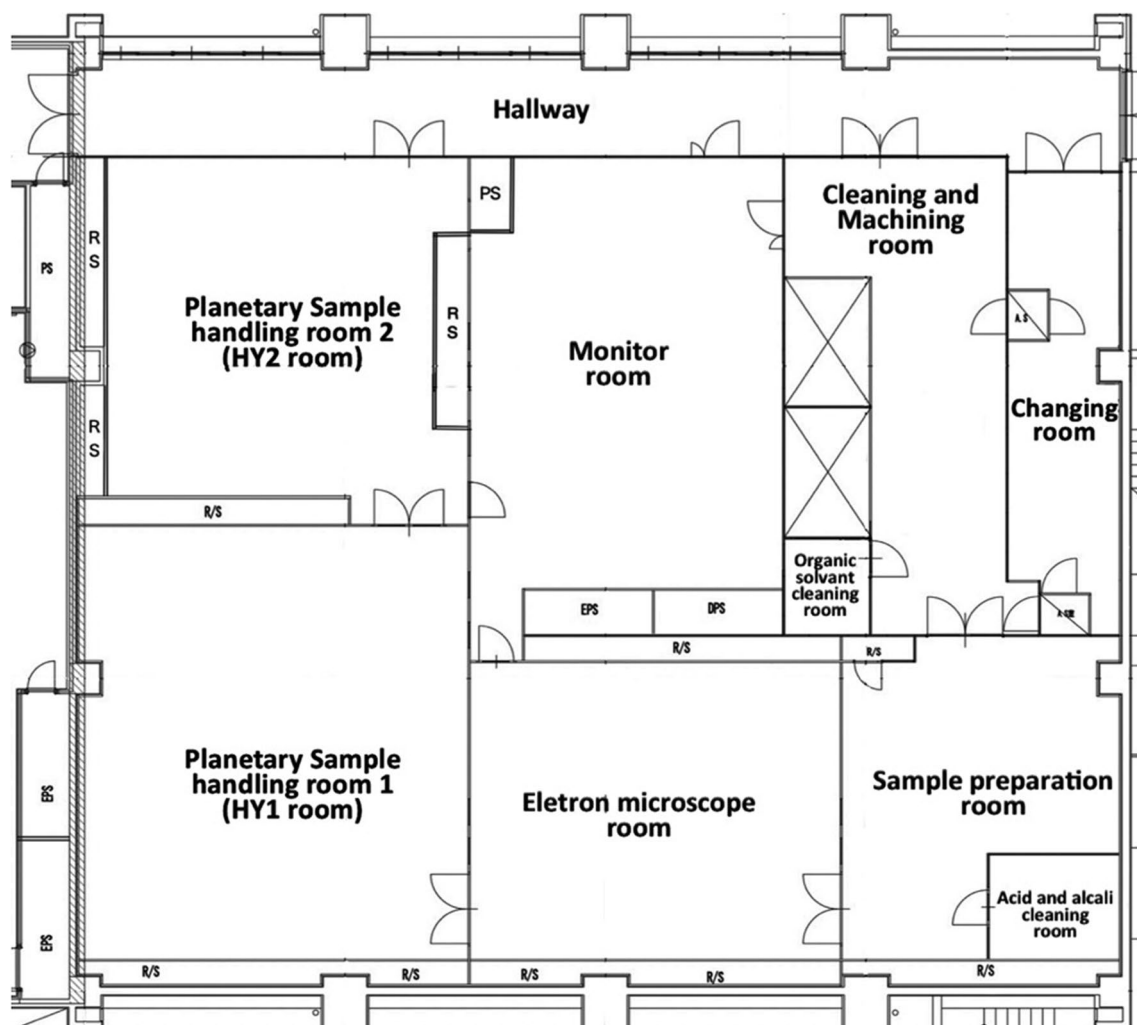


Fig. 1 A schematic of floor plan for clean rooms in the Extraterrestrial Sample Curation Center. The clean room for HY2 samples was newly established in 2017 where clean chambers (CCs) for Hayabusa2-returned samples were installed. RS: a return shaft, PS: a pipes shaft, and EPS: an electronics and pipes shaft

CC3-1 is a vertical tube-shaped chamber with a diameter of 400 mm and a height of 450 mm (Fig. 5a). It is connected to CC3-2 through double ConFlat-305 gate valves and connected to a turbo molecular pump through a gate valve whose back pressure is evacuated with a dry root pump situated in the basement. Typically, CC3-1 reaches less than 5×10^{-6} Pa within this evacuation system. The chamber is also equipped with a pair of getter pumps via gate valves; hence, it can be separated from CC3-2 while keeping vacuum inside. The bottom flange of the chamber is equipped with the container opening system. The container opening system was designed to move the lid of the HY2 sampler container upward from the bottom part of the container fixed to the bottom flange, thus opening the container.

The HY2 sampler catcher with the returned samples inside is attached to the inner lid of the HY2 sampler

container. Thus, as the lid of the container moves upward from its bottom part by the container opening system, the sampler catcher is transferred to the stage 1 of CC3-2 using the transfer rod 1 equipped in the other side of CC3-2, as shown in the side view of CC3-1 in Fig. 5a. After sample catcher transportation to CC3-2, samples spilled down into the bottom part of the container can be observed with a borescope mounted on the side of CC3-1. The borescope is equipped with a straight move-in and -out mechanism and enclosed into a quartz glass tube to observe the inner surface of the bottom part of the container under vacuum conditions. The chamber is equipped with a residual gas analyzer quadrupole mass spectrometer (RGA-QMS) Canon-Anelva M-200TDM that uses an angle valve to monitor gaseous components resided in the vacuum of the chamber. This spectrometer is used for detecting the opening of the sample container.

Table 1 Materials used for the Hayabusa2 clean room and clean chambers

Materials	Use	Remark
Fluorene resin, Barium plate steel	Walls and ceiling of clean room	
Silicon	Caulking agent	
ADC12, Ni–Cr coating	Grating floor	
Epoxy resin	Floor paint	
Soda lime glass	Windows for walls	
Stainless steel 316L EP, Stainless steel 304 BA w/acid cleaning	N ₂ and compressed air gas pipe	
Zinc steel	Local ventilation	
Boron free ULPA	FFU filters for recycling nitrogen purifiers	CC
Stainless steel 304	Clean chambers, vacuum line	CC
A6061	Jigs and tools used for handling samples	CC
Viton	Sealing for sub-chambers and sample containers	CC
Viton-coated butyl	Gloves for clean chambers	CC
Gold	References for IR spectrometer, terminals of wires	CC
PTFE	Electronic insulators, reference material for an optical microscope, jigs	CC
PFA tube	Pipes for nitrogen gas lines of desiccators	CC
Oxygen-free copper	Gasket for clean chambers	CC
Tempered glass	Clean chambers	CC
Quart glass	Viewports, containers for samples	CC
Sapphire glass	Viewports, containers for samples	CC
Nickel–chrome–molybdenum steel	Hex keys	CC
Kapton film	Covering of clean chambers for insulation during baking	CC3-2
Silver solder (Ag, Au, and Zn)	Sampling tool for mm-size grains	CC3-2
PEEK	Connectors for power and signal wires, a pair of tweezers for reference weights	CC4-2
Nylon tube	Pipes for compressed air	
Nytril	Gloves	
Steel plate w/melamine-baking finishing	Cabinets for instruments	
Aluminum foil	Covering of clean chambers for thermal homogenization during baking	
Glass fiber	Insulators for baking heaters	
Acryl resin	Main materials for glove boxes	
Teflon grease	Used for lubrication in clean rooms	Ulvac Super Z300
PVC	Main materials for furniture in acid/alkali cleaning room	
PE	Plastic bags	
PP	Cases for tools and consumables	
Polyester	Clean suits, clean cloths	
Polyurethane	Wheels of cabinets and wagons, soles of clean boots for workers	
Non-woven textile	Wipers, hairnets and masks	

* “CC” indicates materials also used for clean chambers

Typically, residual gaseous species in CC3-1 after the container was opened are H₂O, N₂, O₂, H₂, CH₄, and CO₂.

As CC3-1 is opened to air during the introduction of the HY2 sampler container, the chamber is equipped with nitrogen or dry air purge line on the top of the chamber in order to minimize the adsorption of atmospheric H₂O into the inner surface of the chamber. In case of accidents such as the sample catcher falling inside the chamber, the chamber can be purged with purified nitrogen,

and then, the recycling nitrogen purifier for CC3-3 can be circulated through CC3-2 into CC3-1. Under such circumstances, a glove port can be attached to a gate valve equipped on the side of CC3-1 and then the fallen catcher can be handled and set back into the right position with the glove.

CC3-2 has a width of 550 mm, length of 1140 mm, and height of 400 mm. It is connected with CC3-1 through double gate valves and with CC3-3 through a gate valve (Fig. 5a). The chamber is equipped with three

Table 2 Preparation schedule for the clean room and clean chambers of Hayabusa2 returned samples

Year	2016												2017												2018												2019												2020											
	A	M	J	J	A	S	O	N	D	J	F	M	A	M	J	J	A	S	O	N	D	J	F	M	A	M	J	J	A	S	O	N	D	J	F	M	A	M	J	J	A	S	O	N	D	J	F	M	A	M	J	J	A	S	O	N	D			
Clean room for Hayabusa2																																																												
Preliminary design	←→																																																											
Contract													←→																																															
Detail design													←→																																															
Room construction													←→												←→																																			
Piping installation																									←→												←→																							
Alert system installation																																					←→																							
Clean chamber for Hayabusa2 (CC3-1 and CC3-2)																																																												
Preliminary design	←→																																																											
Contract	←→																																																											
Detail design	←→												←→																																															
Manufacture													←→																																															
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Clean chamber for Hayabusa2 (CC3-3, CC4-1 and CC4-2)																																																												
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Refurbish																																																	←→											

Sample Return Sample Return

sample catcher handling stages and the catcher can be transferred to either of the stages with the transfer rod 2, which is equipped in the shorter side wall of CC3-2, opposite to the CC3-1 side (Fig. 5a). Each of the catcher handling stages allows for X, Y, Z, and theta movement to adjust the position of the catcher to the transfer rods or other equipment for each of the stages. Furthermore, the stages are equipped with a glass viewing port to observe the tools and the catcher. Next, the equipment for each stage is described.

At the stage 1 position of CC3-2, a hex key tool is equipped above the stage to unscrew the hexagon socket head cap screws used to fasten a cover plate of Chamber A of the catcher (Fig. 5b). The tool is movable in X, Y, and Z planes to allow for adjusting the hex key into the hex screw hole. On the longer side wall of CC3-2 (from the back), two different tools are equipped: one is the wobble stick used to grab the top of the screws, and the other is a movable cylindrical container used to store the removed screws. On the longer side wall (from the front), an electrostatic chuck is installed, which can move back and forth and stick to the cover plate of Chamber A of the catcher (made of aluminum alloy A6061; electronically insulated via a Kapton film). An electrostatic chuck can stick plates made of conductive and non-conductive materials with electrostatic force by tuning its charging voltage. It can use voltages in the range of ±1000 V to stick the cover.

Two types of tools are equipped above stage 2: one is a stainless steel grabbing tool for mm-size grains, which can move along the X, Y, and Z axes to bring a specific grain from the catcher into a glass container; and the other is a borescope equipped inside a quartz glass tube that allows for visualizing inside Chamber A of the sample catcher (Fig. 5c). On the longer wall of CC3-2 (from the back), a quartz glass container, which can move back

and forth, is installed to recover and preserve a few grains of mm-size under vacuum conditions. Within the front side wall, a gate valve is installed, which is normally equipped with a glass viewport to observe the catcher and allows for equipping a glove port in case the catcher falls onto the floor of CC3-2 or is stuck on the stage and cannot be removed from there.

The stage 3 harbors a tool used to wipe the surface of a cover plate and remove powder sticking on its surface using a PTFE flipper, which can move along the X, Y, and Z axes (Fig. 5d). The transfer rod 3 is installed within the front side wall and used to place a quartz glass cover on the opening of Chamber A of the catcher and transport the catcher to the next chamber (CC3-3) through the gate valve equipped at the back side wall of CC3-2. CC3-2 is equipped with a turbo molecular pump, which is connected through the floor to a back roughing pump situated in the basement. CC3-2 can reach a vacuum of 10⁻⁷ Pa with this pumping system.

CC3-3 measures 800 mm in length, 500 mm in width, and 600 mm in height (Fig. 2). It is designed to be a vacuum chamber equipped with a turbo molecular pump connected to a roughing pump in the basement. Furthermore, it is equipped with two glove ports with gate valves that can be used to handle tools and jigs under purified nitrogen conditions and atmospheric pressure. Here, the term “jig” refers to a tool developed and used only for a specific purpose. CC3-3 consists of a recycled nitrogen purifier situated in the basement, which is connected to the chamber to circulate nitrogen gas between the chamber and purify the returned gas to be introduced back into the chamber. The pressure within CC3-3 is positively controlled to the atmospheric pressure using the pressure control system installed in the chamber.

CC3-3 contains stage 4, which can move along the X, Y, Z, and theta axes to adjust the position where the

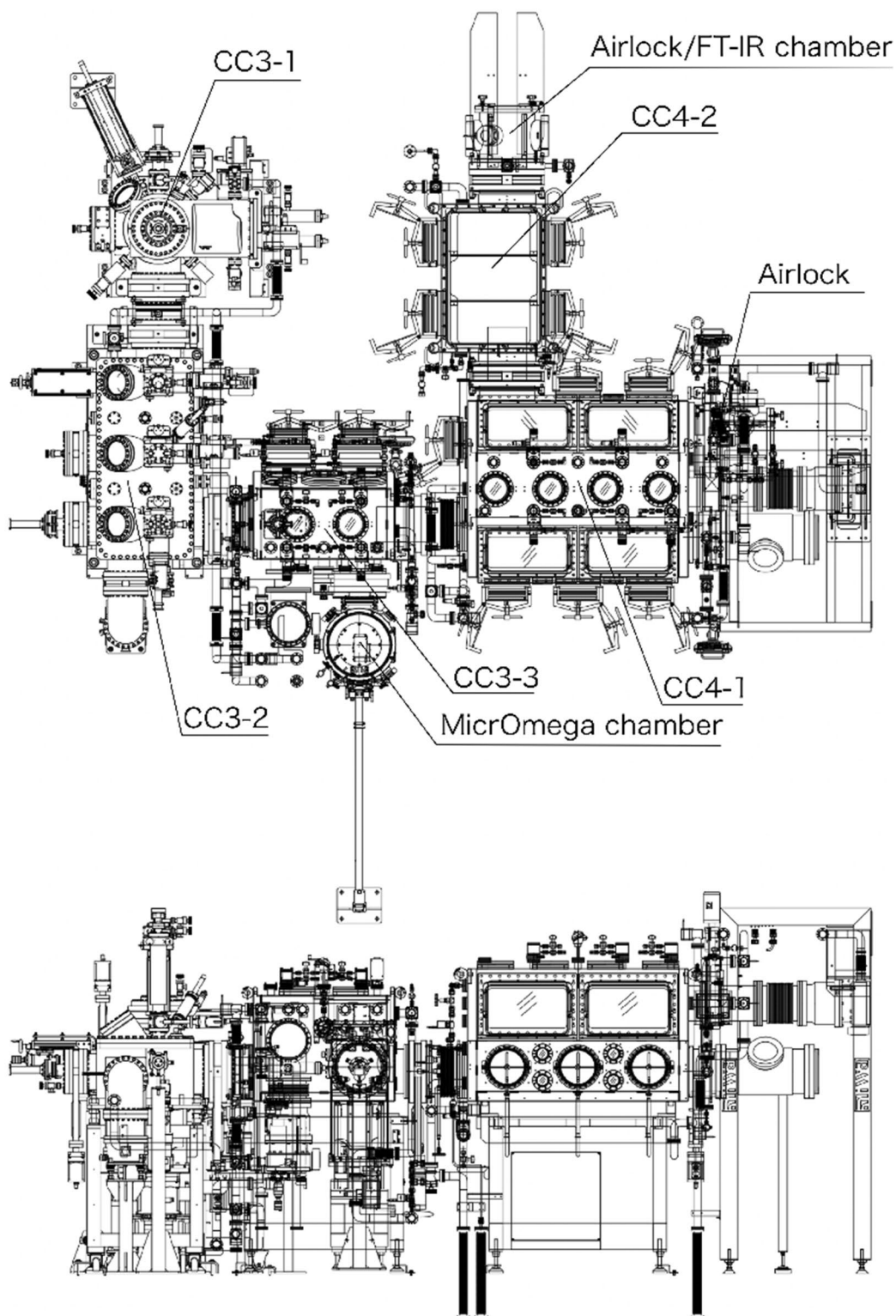


Fig. 2 Detailed schematic of a top view (upper) and a side view (below) of CCs for Hayabusa2 samples. CC3-1 and the CC3-2 are designed for sample processing under vacuum condition, whereas CC3-3, CC4-1, and CC4-2 are designed for sample processing under purified nitrogen condition

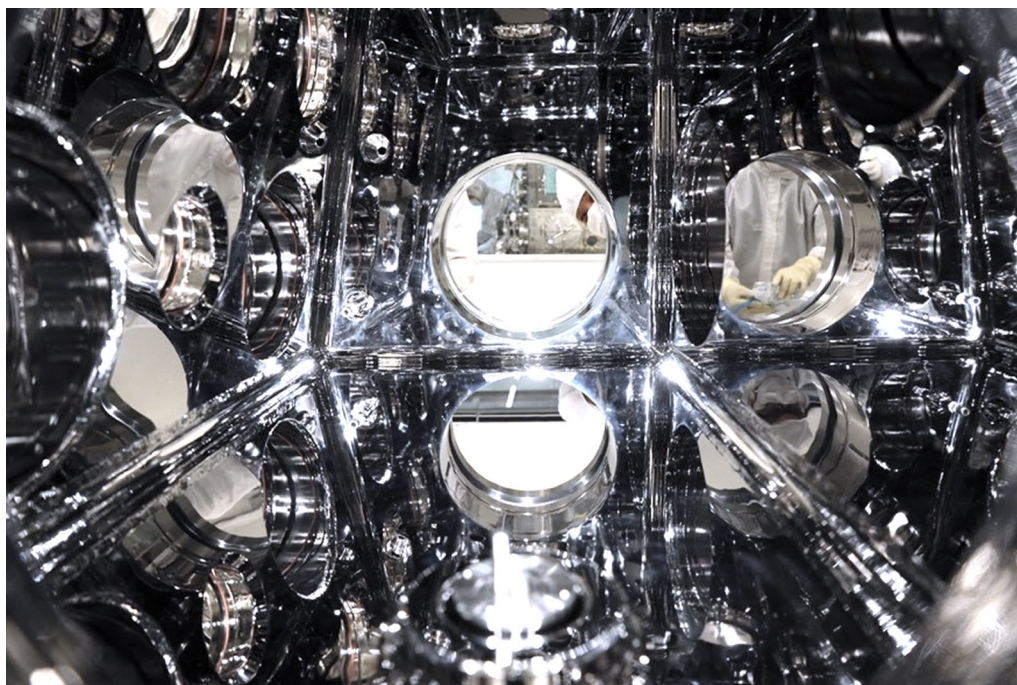


Fig. 3 Image of the interior of the CC3-3. The inner surfaces of all CCs for Hayabusa2 samples have been electrochemically polished like a mirror. This photo was captured during the installation of CC3-3, CC4-1, and CC4-2 at the end of Aug 2018; hence that the chamber was exposed to the air

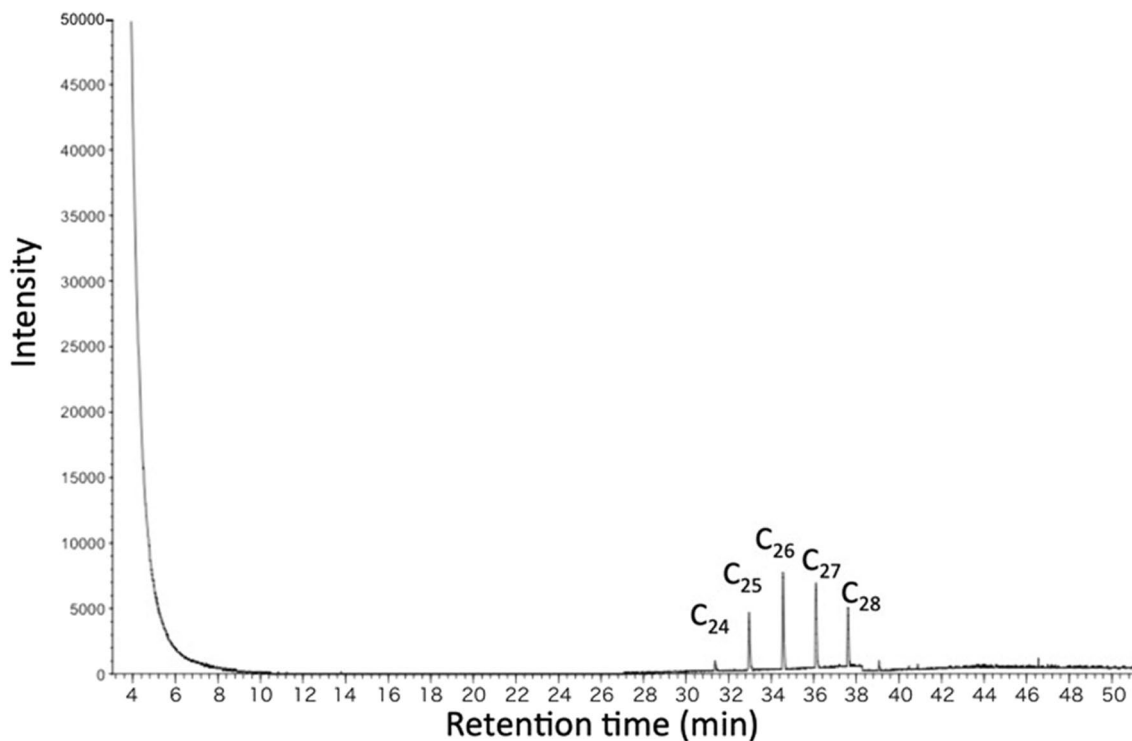


Fig. 4 Mass spectrum of GC-MS obtained from dichloromethane test solution of 2 μ L extracted from a surface of a palm part of Viton-coated butyl glove produced by JUGITEC, a German glove company, shows only a few N-alkane peaks, thus indicating its suitability for handling the Hayabusa2-returned samples

catcher is transported from CC3-2 via the gate valve and receive it into a chamber under vacuum conditions. CC3-3 also has a slow nitrogen purge mode, which allows for the condition of the catcher to be gradually changed from vacuum to atmospheric nitrogen atmosphere after the gate valve between CC3-2 and CC3-3 is closed. The service port with a gate valve is equipped within the back side wall of CC3-3, where an analysis chamber for a MicrOmega (MO), a hyperspectral imager, is situated. CC3-3 also contains a dew-point (DP) meter to measure the concentration of water when the chamber is under the nitrogen condition and is connected to an analysis line for an atmospheric pressure ionization mass spectrometer (API-MS) to measure impurities contained in the nitrogen environment. For handling sample grains and glass containers, CC3-3 harbors a suction line close to the lower corner of the left side wall, which can be connected to a vacuum tweezer. As the storage space in the CCs is limited, stainless steel shelves are installed inside CC3-3. A gate valve to CC4-1 is placed on the side wall opposite to the gate valve to CC3-2. A slide table of 300 mm in length and 200 mm in width is installed on this gate valve to transfer samples, tools, and jigs between CC3-3 and CC4-1.

CC4-1 is 1250 mm in length, 1000 mm in width, and 750 mm in height, with six glove ports on both sides of the chamber. A pair of tempered glass windows of 450 mm in length and 350 mm in width are installed on both sides of the chamber (Fig. 2). An airlock is installed to introduce tools and jigs from outside the chamber, which can be evacuated using the high vacuum and purged with purified nitrogen to exclude terrestrial atmosphere when it is opened to the chamber through a gate valve between them. The airlock harbors baking heaters inside, which can be used to bake metal jigs and tools under vacuum condition and decrease interference gas absorbed on tool surfaces before their introductions to the chamber. Furthermore, CC4-1 is connected through pipes in the floor to a recycling nitrogen purifier situated in the basement. The purifier can circulate nitrogen only for CC4-1 and for both CC4-1 and CC4-2, as CC4-1 is connected to CC4-2 through a gate valve between them.

Slide tables sized 300 mm in length and 200 or 220 mm in width are installed between CC4-1 and CC3-3, as well as between CC4-2 and the airlock for safe transfer of samples, tools, and jigs. The pressure of CC4-1 is positively controlled to the atmospheric pressure with the pressure control system installed within the chamber. CC4-1 also harbors a DP meter and an analysis line for API-MS, which allows for monitoring impurities in nitrogen down to sub-ppb level. CC4-1 is the most spacious chamber among the CCs for HY2; thus, it is used for disassembling the sample catcher and extracting samples onto sapphire dishes. Additionally, it is used for storage of individual Ryugu grains and bulk (aggregate) Ryugu samples under purified nitrogen conditions. For handling sample grains and the glass containers for samples, CC4-1 is equipped with a suction line, which can be connected to a vacuum tweezer that allows for handling mm-sized grains and cover plates of the sample containers. For cases when samples <0.1 mm would require handling, an electrostatically controlled micromanipulation system can be installed in CC4-1, which is identical to that installed in CC2 for handling <0.1 mm Itokawa grains returned by HY1 in 2010 (Yada et al. 2014, 2022b).

CC4-2 is 840 mm in length, 540 mm in width, and 346 mm in height (Fig. 2). CC4-2 is equipped with four glove ports, a pair of gloves from its frontside where the optical microscope is located, and another pair of gloves from the opposite side. CC4-2 is connected to CC4-1 through a gate valve where nitrogen flow down from CC4-1 returns to the recycling nitrogen purifier situated beneath the floor in the basement and also contains a slide table of 300 mm in length and 220 mm in width for smooth transportation of samples, jigs, and tools between CC4-1 and CC4-2. CC4-2 can be high vacuum evacuated using the pumping system of CC4-1 through the gate valve between them. On the other side of CC4-1, there is another gate valve that allows the connection of a Fourier Transform Infrared spectrometer (FT-IR) analysis chamber or an airlock. CC4-2 contains its own pressure control system to keep the inner pressure positive to the atmospheric pressure. It harbors a suction line that can be connected to a vacuum tweezer to enable the handling of mm-sized grains and cover plates of the sample containers. The other service port can be used as

(See figure on next page.)

Fig. 5 Schematics of the CCs for Hayabusa2-returned samples. **a** A cross section of a side view of CC3-1 and CC3-2. CC3-1 is equipped with a container opening system. The container opened in CC3-1 under vacuum would be transferred to the CC3-2. The CC3-2 has three container handling stages to open and remove the cover of the chamber A of the sample catcher, remove a few grains inside the chamber A of the catcher, and transfer the catcher with the remaining samples to CC3-3. **b** A cross section at the position of the stage 1. A cover plate of the chamber A of the catcher was opened at this stage. **c** A cross section at the position of the stage 2. A few mm-sized grains inside the chamber A of the catcher were removed at this stage. **d** A cross section at the position of the stage 3. The catcher including samples was transported to CC3-3 from this stage

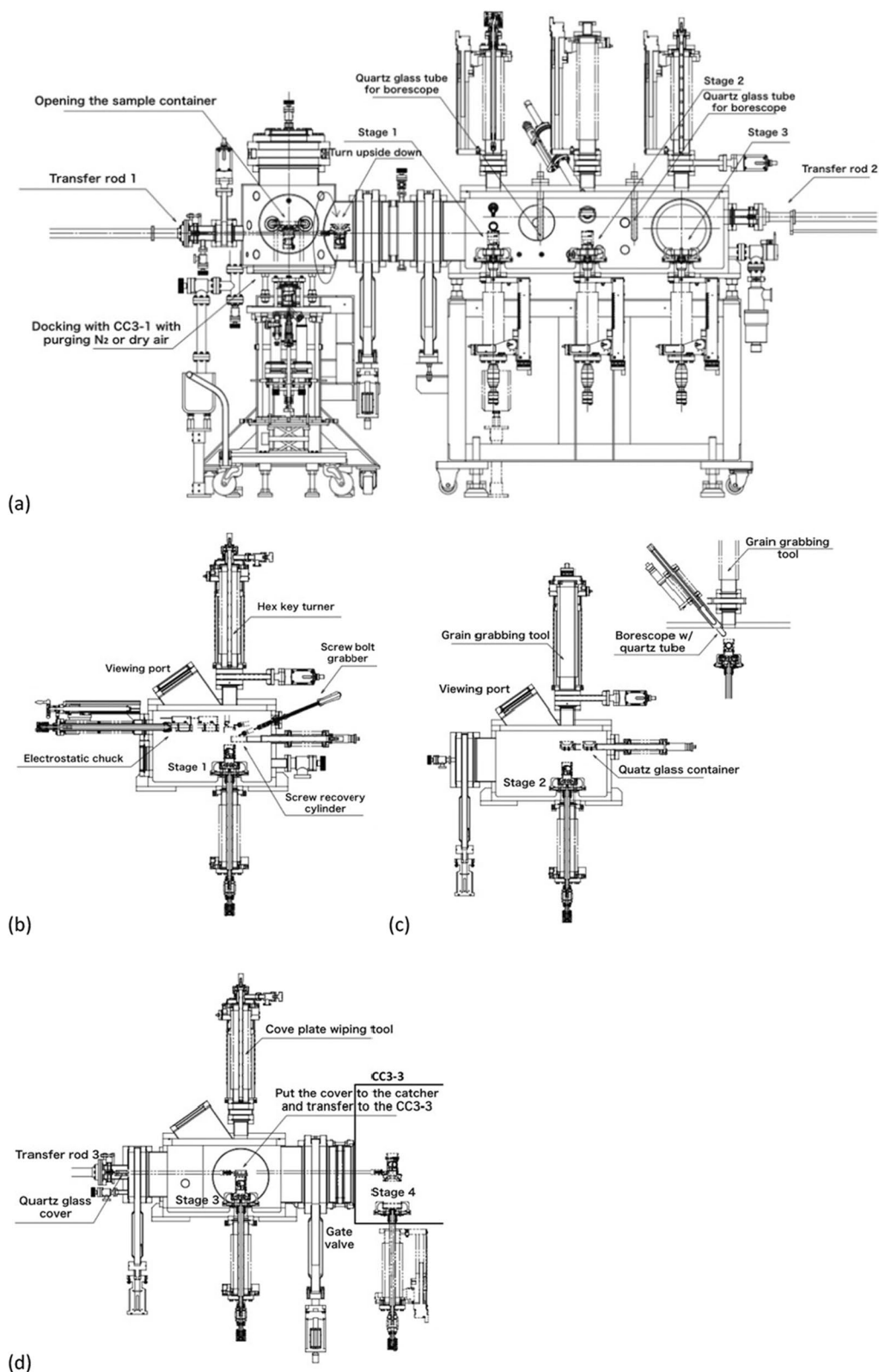


Fig. 5 (See legend on previous page.)

a hermetic feed through port for connecting signal and power supply cables to a balance inside the chamber. As this chamber has a horizontal tempered glass window of 740 mm in length, 490 mm in width, and 12 mm thick on the top side, an optical microscope for sample observation and a monochronic digital microscope for visible reflectance spectrum analyses can be set above the chamber.

Three additional chambers are present: an MO chamber connected to CC3-3, a storage chamber connected to CC4-1, and an FT-IR chamber connected to CC4-2. The MO chamber is composed of two chambers: a sample chamber made of stainless steel 304, connected through the gate valve to the back side of CC3-3; and an instrument chamber made of stainless steel 304 base and an acrylic cover placed immediately above the sample chamber. The sample chamber has several components: electronic moving sample stage for X, Y, Z, and theta axis motion; a Peltier device cooler to cool the stage down to $-5\text{ }^{\circ}\text{C}$ for decreasing thermal noise from samples during analysis; and a transfer rod to introduce a sample holder from CC3-3 to the sample stage. The cylindrical shaped sample chamber (254 mm in diameter and 310 mm in height) is vertically set up and the glass viewport of ConFlat flange (203 mm in diameter) mounted on its side is used to adjust the position of the sample holder handled using the transfer rod. This is fitted within another holder on the sample moving stage. A sapphire glass viewport of ConFlat flange (114 mm in diameter) is inserted between the instrument and the sample chamber, which allows for the MO analysis of the samples on the sample stage. The MO sample chamber can be high vacuum evacuated, typically less than 10^{-3} Pa, through the vacuum line and also purged with purified nitrogen at atmospheric pressure through the nitrogen gas purge line. The instrument chamber is 252 mm in diameter and 200 mm in height and attached on a copper plate cooled down to $10\text{ }^{\circ}\text{C}$ with a coolant water circulated, with a chiller situated in the basement. The instrument chamber is purged with purified nitrogen to avoid dew condensation onto the MO.

The storage chamber is attached to the service port located adjacent to CC4-1 airlock through a gate valve. The chamber is horizontally oriented and cylindrically shaped (640 mm in length and 250 mm in diameter). The chamber is equipped with a slide table (550 mm in length and 200 mm in width) designed for the transport of samples into the chamber for storage. It can be high vacuum evacuated, typically less than 10^{-3} Pa, using the pumping system from CC4-1 after closing the gate valve adjacent to CC4-1 and purged with purified nitrogen at atmospheric pressure.

The FT-IR chamber is 300 mm in length, 150 mm in width, and 190 mm in height and equipped with an

electronic X-Y moving sample stage. A ConFlat flange of a sapphire viewport, recently replaced with a CaF_2 viewport of 152 mm in diameter, is placed on top of the chamber. This chamber allows for sample analysis on the moving stage inside the chamber without exposing them to air, using the FT-IR JASCO VIR-300. Similar to the airlock compatible to the FT-IR chamber, this chamber can be high vacuum evacuated, typically less than 10^{-3} Pa, with an independent pumping system equipped in the HY2 sample handling room and purged with purified nitrogen at atmospheric pressure.

It is important to note that all containers, jigs, and tools installed into the CCs are cleaned with a protocol established in the Extraterrestrial Sample Curation Center (ESCuC), which is basically composed of degrease cleanings—a series of ultra-pure-water overflow cleanings with ultrasonic cleaners at 35 kHz, 95 kHz, and 950 kHz plus UV ozone cleaning and/or alkali solution cleaning (Yoshitake et al. 2021).

Contamination control of cleanrooms and CCs and installation schedule

We aimed to process the samples under the least amount of contamination; therefore, we assessed the contamination from the major procedural stages ranging from pre-launched to sample distribution. The detailed contamination control procedure of the HY2 sampler in the pre-launched phase is described by Sakamoto et al. (2022). Contaminations from inorganics and organics in clean rooms and CCs for HY2 samples are periodically tested (at least once a year) as described by Yoshitake et al. (2021) and Hitomi et al. (2023). Here, we describe the contamination control of cleanrooms and CCs during preparation for receiving samples and after the sample return. Figure 6 shows (a) organics, (b) negative and positive ions, and (c) metal element abundances in the cleanrooms and CCs throughout the last 4 years (from 2018 to 2022). Organics and metal elements concentrations were examined using the Si wafer deposition method. P type Si wafers of 200 mm in diameter were exposed to the environment for 15–20 h and sent to an analysis contractor to be measured using a thermal decomposition GC–MS (TD-GC–MS) and a vapor phase decomposition inductively coupled plasma mass spectrometer (VPD-ICP-MS) (Yoshitake et al. 2021). Positive and negative ions concentrations were only examined under atmospheric conditions in cleanrooms using the impinger method (Yoshitake et al. 2021). Compared with the HY1 sample room, the HY2 sample room contains two to six times higher concentrations of total organic compounds (Fig. 6a). However, the concentration has been decreasing over the years as the vaporization of contaminants from the new construction decreased. The contaminants

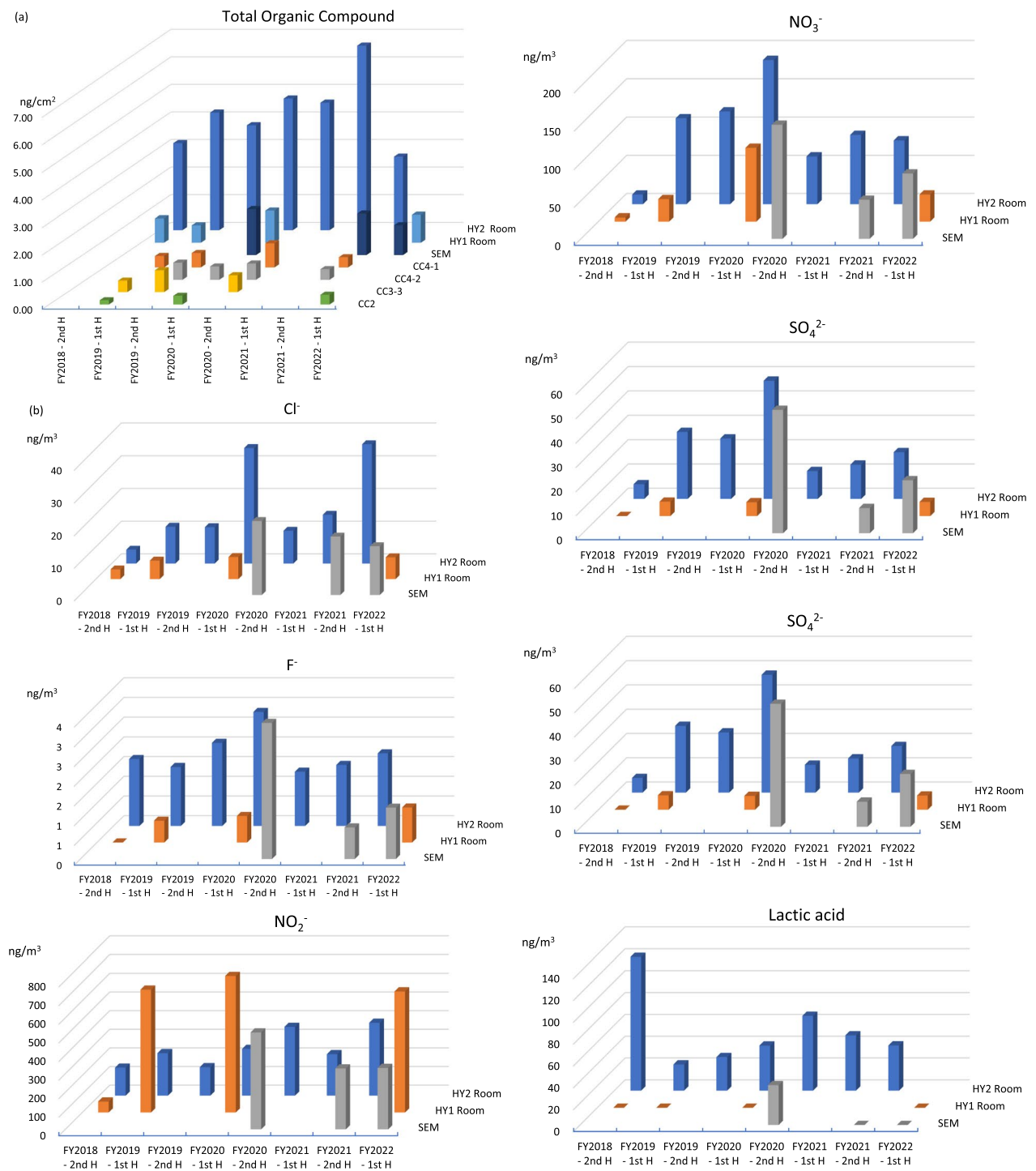


Fig. 6 Concentrations of **a** organics (ng/cm²), **b** negative and positive ions (ng/m³) and **c** metallic elements (atoms/cm²) detected in the clean rooms and/or the CCs (CC2, CC3-3, CC4-1 and CC4-2). **a, c** Analysis of contaminants deposited on surfaces of Si wafers exposed to the environments for 15–20 h using GC–MS and ICP–MS, respectively. **b** Contaminants in cleanrooms assessed using the impinger method and analyzed using ICP–MS

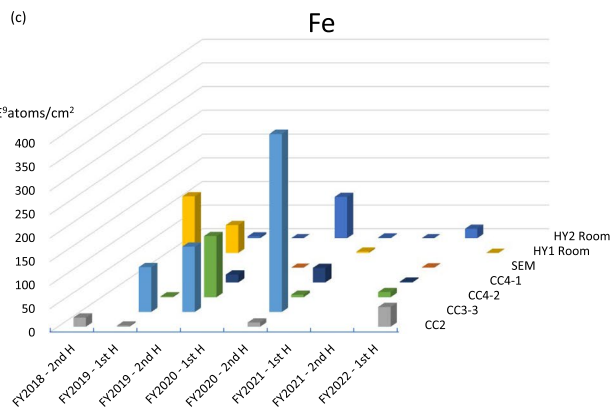
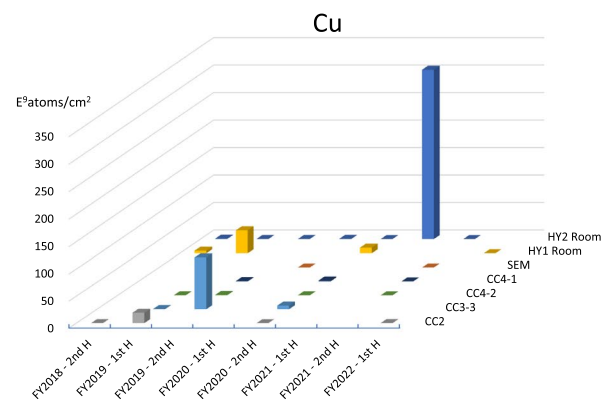
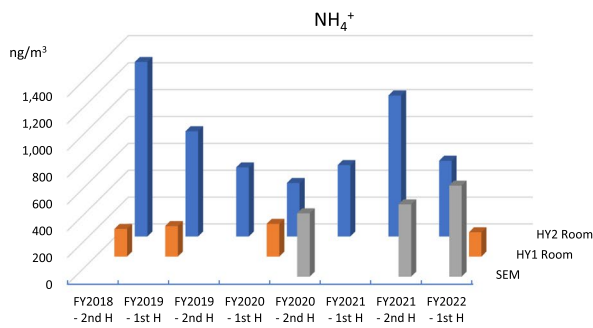
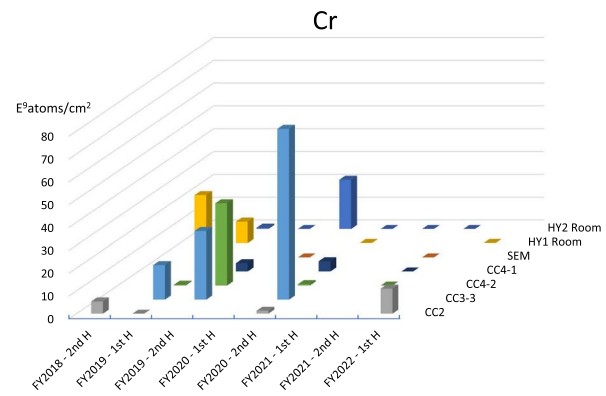
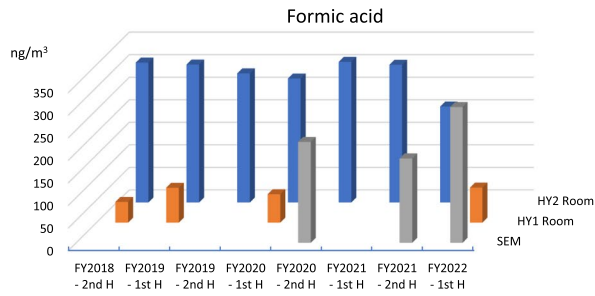
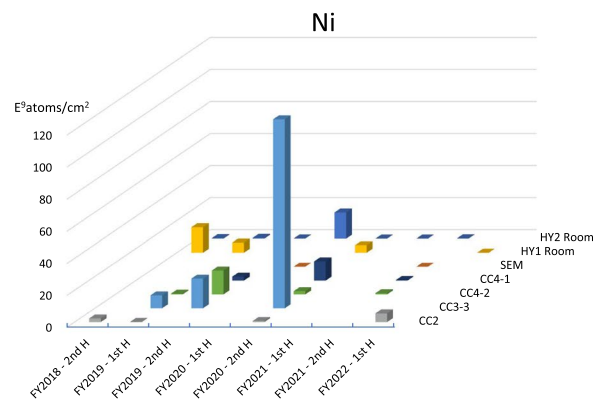
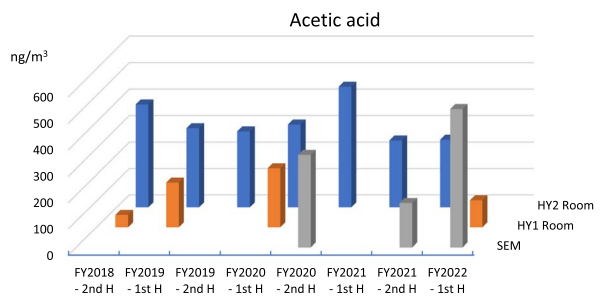


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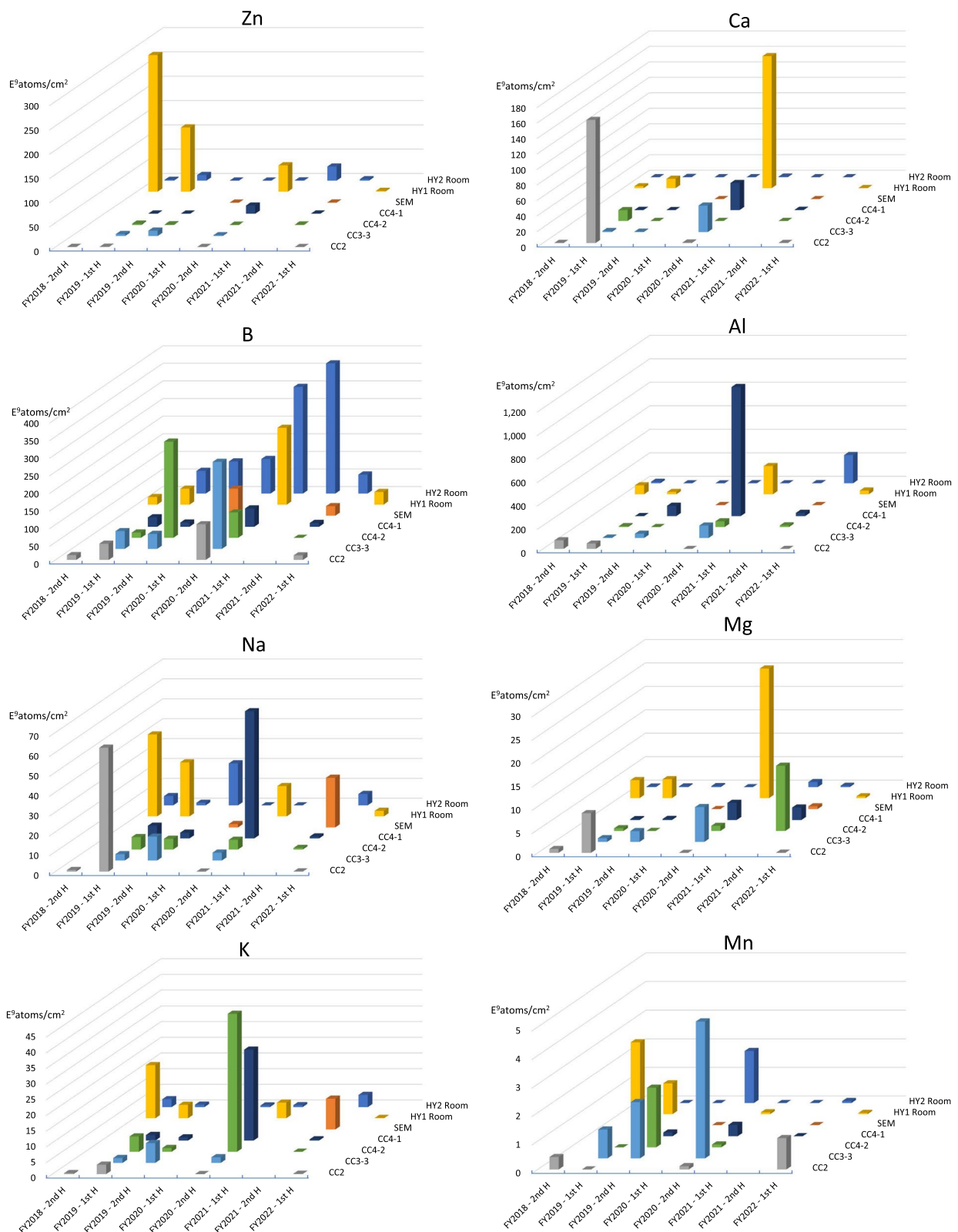


Fig. 6 continued

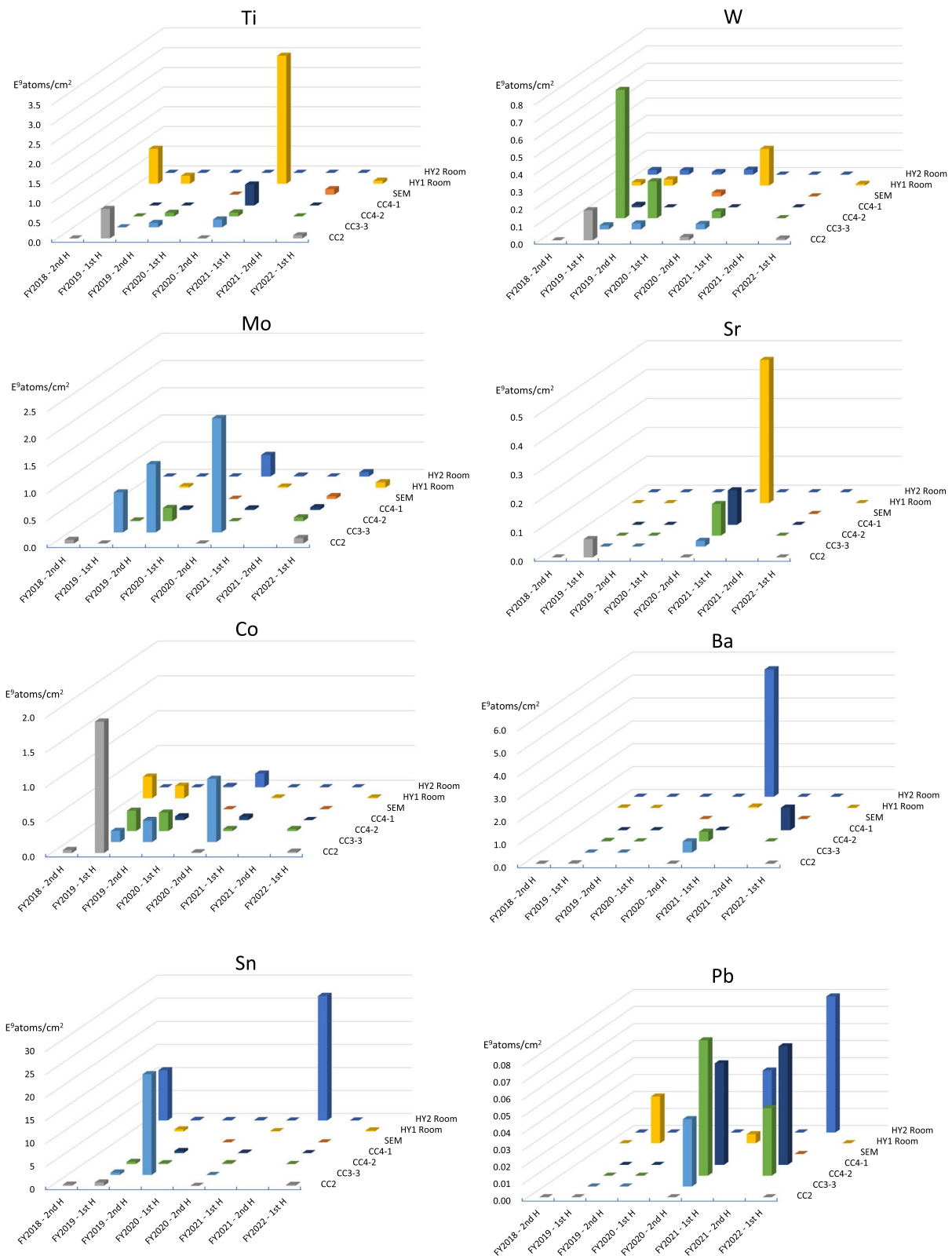


Fig. 6 continued

in the CCs were lower than 1 ng/cm^2 , which is sufficient for receiving returned samples and much lower than the Stardust amino acid abundance reported to be 186 ng/cm^2 (Dworkin et al. 2018).

The concentrations of 32 metal elements (Ag, Al, Au, B, Ba, Ca, Cd, Co, Cr, Cu, Fe, Ga, Hf, In, K, La, Mg, Mn, Mo, Na, Ni, Pb, Pd, Pt, Sn, Sr, Ta, Ti, W, Y, Zn, and Zr) were examined with detection limits of less than 1×10^9 atoms/cm². Abundances of Ga, Y, Pd, Ag, Cd, In, Hf, Ta, Pt, and Au in the HY2 room were lower than their detection limits in most cases, and thus, they are not shown or discussed in this study. Relatively large amounts of Fe, Cr, and Ni were detected, mainly in the HY2 CCs (CC3-3, 4-1, and 4-2), although a decrease in content to less than 10^{10} atoms/cm² was measured after 2021 (Fig. 6c). As these elements are primary components of the CCs, we cannot exclude their ability to contaminate the CCs. Concentrations of lithophile elements, such as B, Na, Mg, Al, K, and Ca are indicators of terrestrial detritus grains contamination. Concentration of B was once detected at $\sim 2 \times 10^{11}$ atoms/cm² in CC3-3 during the second half of 2020; however, recently, its content decreased to less than 10^{10} atoms/cm². Na and K were once detected at $\sim 5 \times 10^{11}$ atoms/cm² in CC4-1 and CC4-2 during the second half of 2020, while in 2021, the content decreased to less than 10^{10} atoms/cm². The concentration of Ca remained lower than 3×10^{10} atoms/cm² after the installation procedures. Aluminum reached 10^{12} atoms/cm² in CC4-1 in 2020. As aluminum is one of most frequently used materials for jigs and tools in the CCs and has a lower hardness than stainless steel 304, it should raise dust; however, its concentration decreased to less than 10^{11} atoms/cm² in all CCs in 2021. Concentrations of Mg and Mn remained lower than 10^{10} atoms/cm² after the installation procedures. The concentration of Ti, Co, Sr, Mo, and Ba remained lower than 10^9 atoms/cm² after the installation procedures. The concentration of Pb remained lower than 10^8 atoms/cm² throughout the years after the installation procedures.

Figure 7 shows profiles of degrees of vacuum in CC3-1 and CC3-2 since 2019. These CCs have been maintained under vacuum in the range of 10^{-6} Pa, occasionally reaching 10^{-7} Pa after baking. In 2019, three rehearsals were performed to confirm the workflow for receiving the HY2-returned sampler container, shown as three peaks of high pressures (Fig. 7). Regular rehearsals were conducted periodically in 2020 before receiving the container, and during rehearsals, the pressures of CC3-1 and CC3-2 remained lower than 2×10^{-6} Pa. The pressures of CC3-1 and CC3-2 have been maintained under 5×10^{-6} Pa and 1×10^{-6} Pa, respectively, after receiving the container. Thus, the vacuum within CC3-1 and CC3-2 has

been maintained low enough to preserve small portions of samples.

CC3-3, CC4-1, and CC4-2 have been monitored using the API-MS (APIMS-200 produced by Nippon API Co. Ltd) that can analyze impurities in nitrogen gas at atmospheric pressure at sub-ppb level (Irie et al. 1995). As the API-MS can analyze only one environment, we switched the introduction lines between CC3-3, CC4-1, and CC4-2 to measure each environment. The API-MS was calibrated for H₂O, O₂, CO₂, and CH₄ concentrations using their standard gases once a year. We measured the concentrations of interferent molecular gases in the nitrogen environment of CC3-3, CC4-1, and CC4-2 before receiving the samples. Figure 8a shows impurity concentrations in the nitrogen environment of CC3-3 and CC4-1 in August 2020. All impurity concentrations were maintained under 200 ppb throughout the month of August. Figure 8b shows impurity concentrations in CC4-2 in March 2021. Although the HY2-returned samples were frequently treated in March 2021, H₂O and O₂ were maintained at levels under 200 ppb and CO₂ as well as CH₄ were at levels under 10 ppb in CC4-2. Figure 8c shows the impurity concentrations in CC4-1 in March 2022. As the purified nitrogen flow in CC4-1 is upstream of CC4-2, the concentration of O₂ is half an order of magnitude lower than that in CC4-2 (Fig. 8b). The H₂O concentration in CC4-1 ranged between 100 and 200 ppb, nearly comparable to that in the CC4-2 (Fig. 8b). Therefore, the purified nitrogen conditions in CC3-3, CC4-1, and CC4-2 were adequate for handling the HY2-returned samples.

We conducted microbial contaminant tests in each glove, the floor of CC4-1 and CC4-2, and the grating floor of the room on November 30, 2021. During the tests, all the HY2-returned samples were temporarily moved to CC3-3, to minimize the risk of contamination during the microbial contaminant test. Encapsulated dry swabs were introduced into CC4-1 and CC4-2. We used the swabs to wipe 300 cm² of area for the inner floor of CC4-1 and CC4-2 and a surface of a Viton-coated butyl glove from CC4-1. Similarly, the surface of the aluminum grating floor of the planetary sample handling room 2 (HY2 sample room) was also wiped using a swab. The wiped swabs were immersed in 15 mL of phosphate buffered saline (PBS) solutions and mixed using a vortex. Next, 2-8 mL of PBS solution was dropped inoculated onto different types of agar plates and the microbial cells were incubated for 7 days. Figure 9 shows the incubation results on (a) tryptic soy agar plates, (b) blood agar plates, (c) Reasoner's 2 agar (R2A) plates, (d) potato dextrose agar plates, (e) Sabouraud-dextrose agar plates, (f) Sabouraud-dextrose with chloramphenicol agar plates, and (g) thioglycolate plates for each of the PBS solutions.

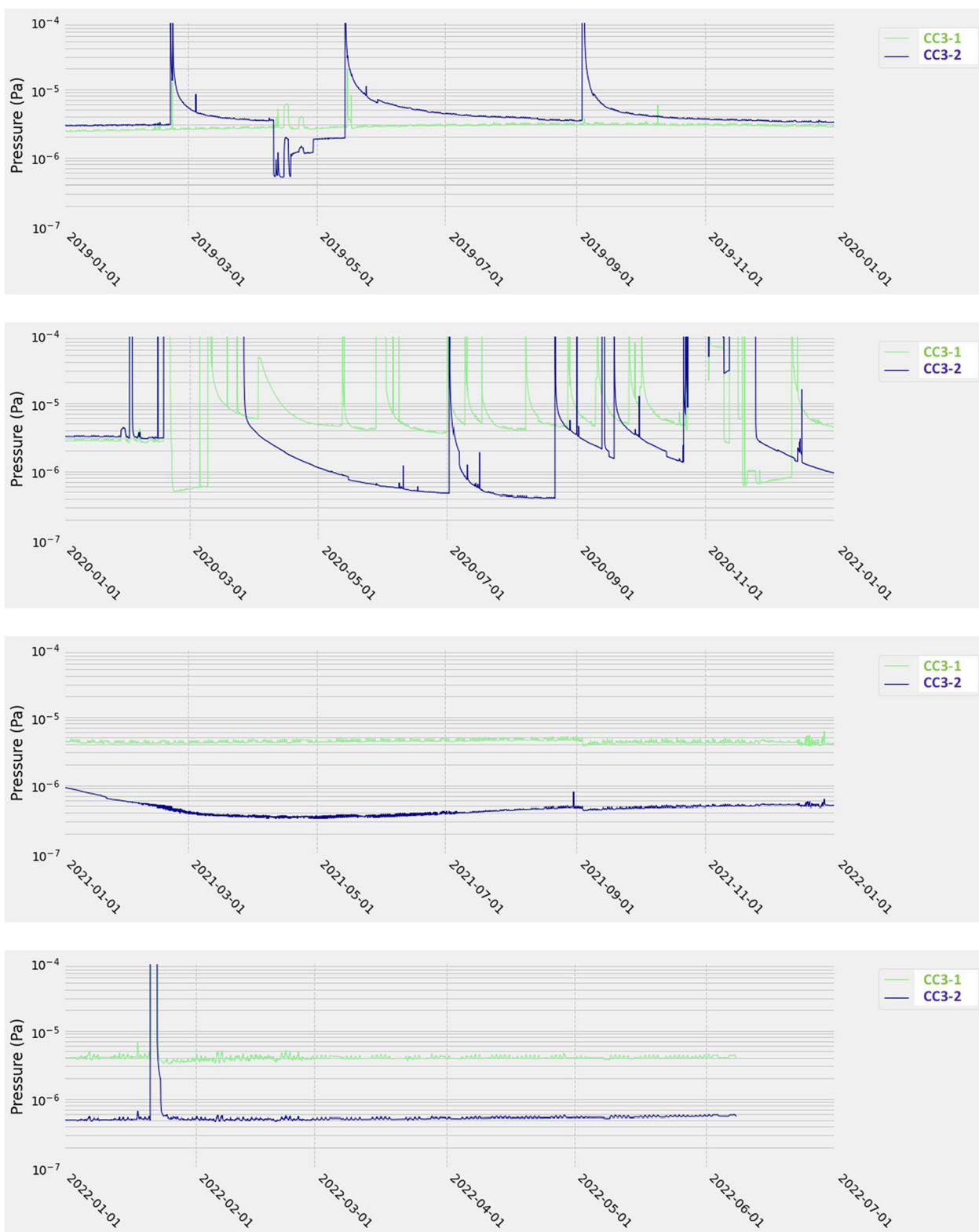


Fig. 7 Profiles of degrees of vacuum in CC3-1 and CC3-2 since 2019. During the rehearsal for introducing a sample container in CC3-1, the CC3-1 has been frequently purged to the atmospheric pressure from 2019 to the end of 2020. The atmospheric purge of CC3-1 in December 2020, when the Hayabusa2 sampler container was introduced into the chamber. Since then, both of the CCs have been maintained under vacuum level lower than 5×10^{-6} Pa, except for lower January 2022 (lower levels), when the planned electronic outage was conducted and all the valves of the chambers were closed under the vacuum conditions

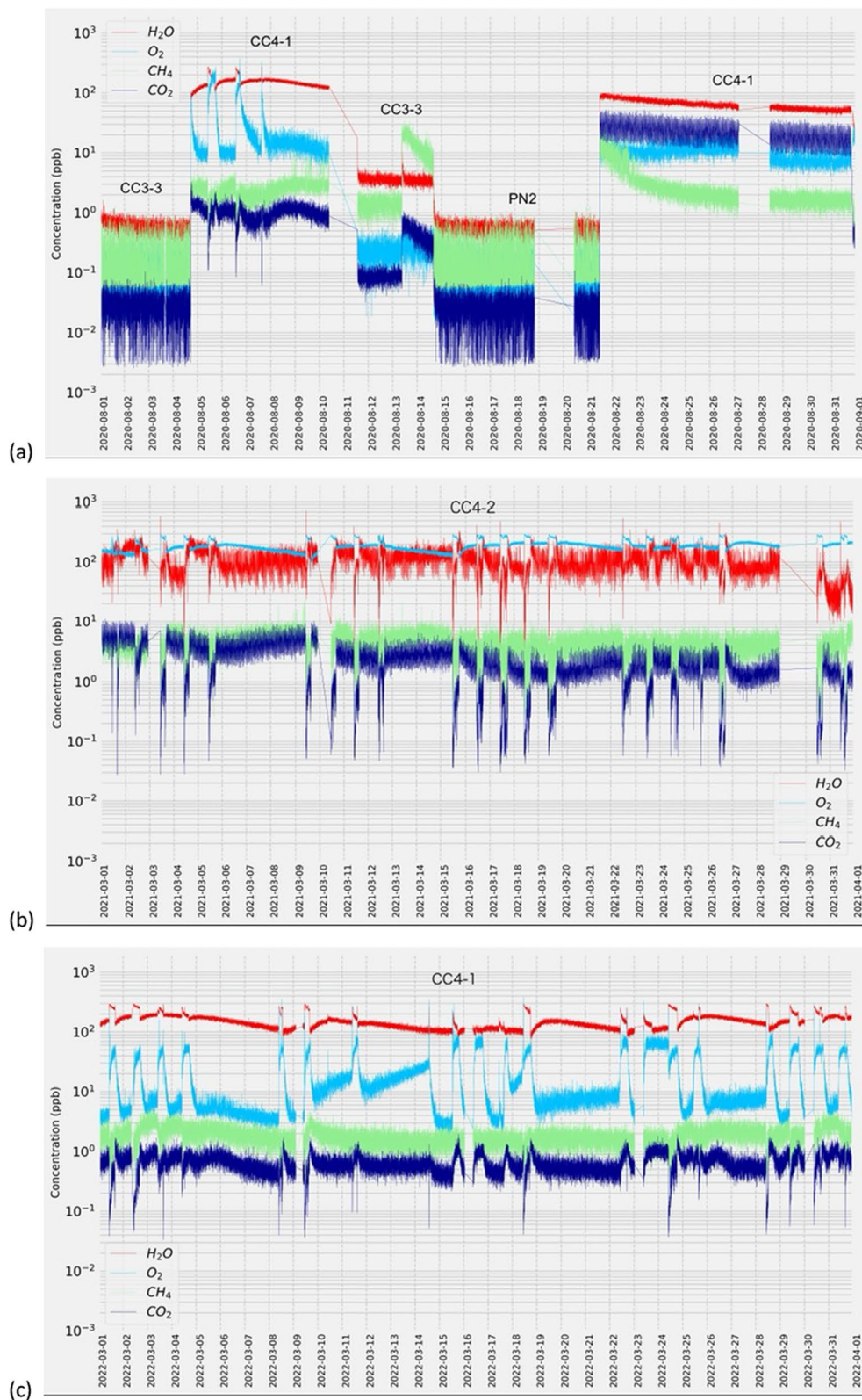


Fig. 8 Impurity concentrations in the nitrogen environment of CC3-3, the CC4-1 and/or CC4-2 analyzed using the API-MS in **a** August 2020, **b** March 2021, and **c** March 2022. The impurity concentrations were lower than 200 ppb

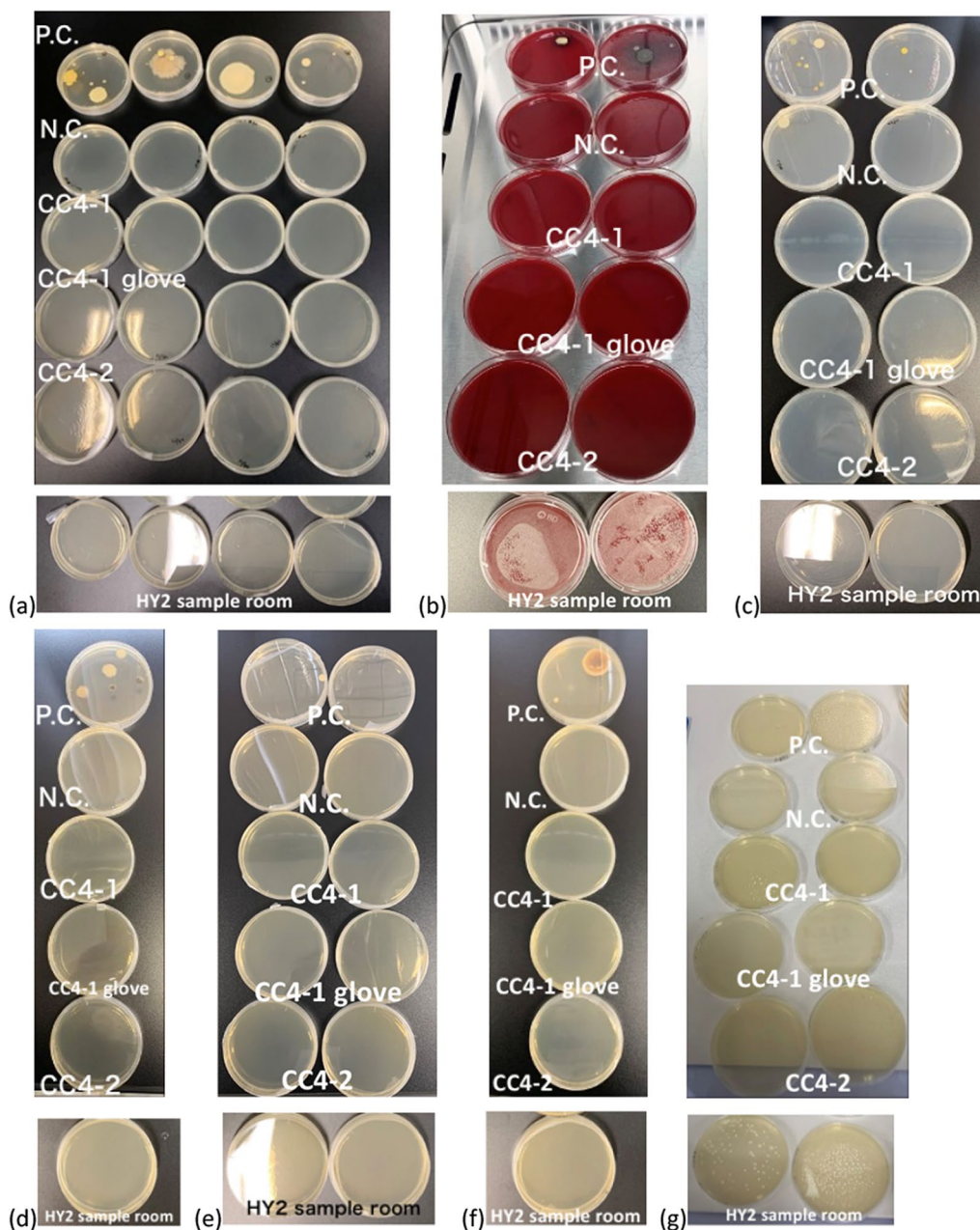


Fig. 9 Results of microbial testing for CC4-1, CC4-1 glove, CC4-2, and HY2 sample room. The incubation on **a** tryptic soy agar plates, **b** blood agar plates, **c** reasoner's 2 agar (R2A) plates, **d** potato dextrose agar plates, **e** Sabouraud-dextrose agar plates, **f** Sabouraud-dextrose with chloramphenicol agar plates, and **g** thioglycolate plates for each of the wiped swabs are displayed. The positive control plate shows multiple colonies on, whereas none of CC4-1 and CC4-2 floor or CC4-1 glove and floor of Hayabusa2 sample room shows any colony

Note that the Sabouraud-dextrose with chloramphenicol agar plates (Fig. 9f) were the only plates incubated in anaerobic condition to confirm the contamination of anaerobic microbes. The results showed that colonies were only present on the positive control plates. This confirms the cleanliness of these environments from a microbial point of view.

Handling tools for the HY2-returned samples

Optical microscopic imaging was conducted in CC4-2 through a tempered glass window. For clear microscopic imaging of each grain, a customized sample stage was set up inside of CC4-2 (Fig. 10). As there is no tapped hole on the inside floor of CC4-2, a series of base plates with tapped holes of 5 mm in thickness was secured onto the

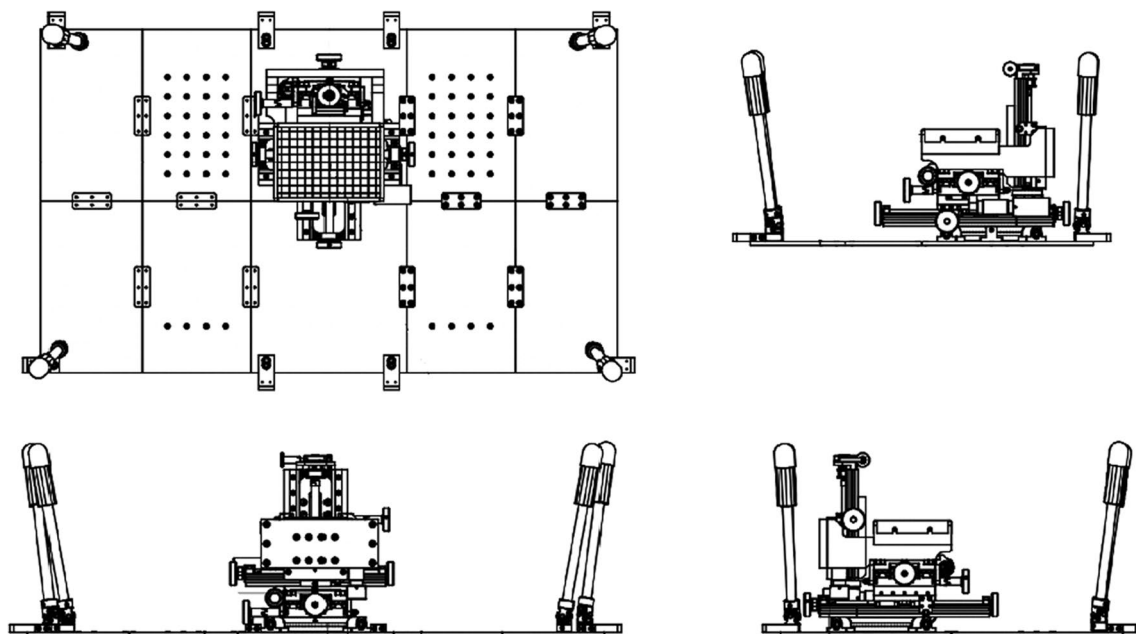


Fig. 10 A sample moving stage in the CC4-2. It is composed of a series of base plates, four shafts, and a sample moving stage capable of moving along the X, Y, Z, and theta axes

corners of the floor using four elastic shafts. An X, Y, Z, and theta moving sample stage was set on the base plates. Its top plate was 131 mm^2 in size, and was movable along the X and Y axes ($\pm 52 \text{ mm}$ and $\pm 73.5 \text{ mm}$, respectively). The travel distance of the plate along the Z axis was 64.5 mm , and 360° along the theta axis. The stage can be moved along X, Y, and Z axes with a coarse or micro-adjustment knob (for micro-motion knobs). Most components of the stage were made of stainless steel 304 and aluminum alloy A6061, although insulators consisting of PTFE were used as part of the stage. The sample stage uses the same design as the one installed in CC-2 for Itokawa samples, except its size and range of motion are different (Yada et al. 2022b). The stage was mostly operated manually using the gloves of CC4-2, turning the knobs of the moving stage to position the samples for imaging.

A vacuum tweezer, which is the most frequently used tool to handle grains of samples, is a commercial product, F002; Fluoro Mechanic Co., Ltd, Edogawa, Tokyo, Japan (Fig. 11a). We customized the anterior portion of the original tweezer to better suit the small-particle processing without contamination. The customized portion consists of two removable attachments: a needle connected to a nozzle, and a connector to a PFA tube (Fig. 11a). Its original nozzle tip was replaced with a thin needle nozzle made of stainless steel 304 (Hamilton Company), removable via a small knurled connector; 30 mm long, 90-degree-cut point style needles were attached.

Their gauge sizes (#24, #21, and #18) correspond to the inner diameters of 0.311, 0.514, and 0.838 mm, respectively, and they were used in accordance with the grain size, such as 1–2 mm for 0.311 mm nozzle, 2–5 mm for 0.514 mm nozzle, and $>5 \text{ mm}$ for 0.838 mm nozzle. The original tube connector made of PTFE was replaced with one made of PFA (6 mm in the outer diameter), which was used for connecting the tweezer to the suction port equipped in CC3-3, CC4-1, and CC4-2. A small portion of the PTFE membrane filter, WINTEC PMF-500, $5 \mu\text{m}$ pore size and $110 \mu\text{m}$ thickness, was set inside the upper joint of the vacuum tweezer to recover powdery samples that may have been aspirated through the tube nozzle. Each of the suction lines of the CCs were connected to the independent pumping system and pumped with a roughing pump to reach $<1 \text{ Pa}$. The suction line was equipped with a pair of stop valves, a needle valve, and a pneumatic valve controlled by a foot pedal for CC4-2 or a button on a touch panel controller for CC3-3 and CC4-1. To avoid contamination and damaging the tip of the stainless needle nozzle, the needle nozzles were stored with a cylindrical-shaped cover made of stainless steel 304.

To handle small grains of $<0.5 \text{ mm}$ in all three dimensions, looped stainless steel needles were prepared (Fig. 11b). The shape of the loop of the needle was determined through repeated tests using grain simulant by multiple processors. The size of handled grains depends on the size of the loop; thus, stainless needles of different

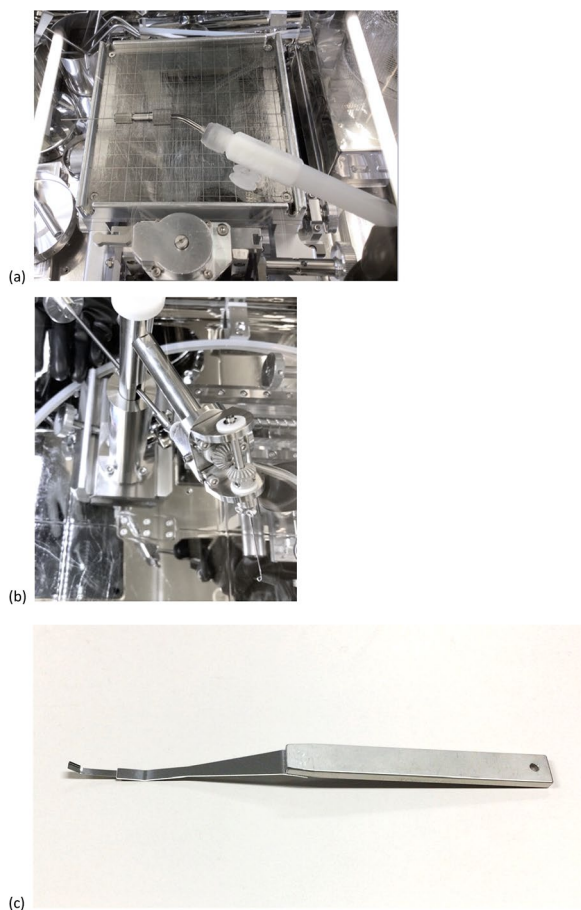


Fig. 11 Image of **a** a vacuum tweezer, **b** a looped needle, and **c** a spatula used for handling sample grains inside the CCs. Every tool was cleaned using a method of a series of cleaning protocols conducted in the ESCuC (Yoshitake et al. 2021) and also demagnetized with a demagnetization instrument, Hozan HC-31, before being introduced in the CCs

diameters were prepared for handling various sizes of sample grains. The needle stand was developed for locking the position of the looped portion of the needle under the optical microscope and rotating the needle to release grains into a container. In handling a grain with the looped needle, the position of the needle was locked with the stand while the grain on the stage approached the looped needle by moving the sample stage along the X, Y, and Z axes with coarse- or micro-adjustment. After the grain was placed on the looped needle, the needle was rotated by the needle stand to release the grain into the container. A cover made of stainless steel 304 was prepared for the looped needle to avoid contamination and needle damage.

We treated samples smaller than 1 mm as an “aggregate” of sample grains, rather than one by one. Thus, a tool to scoop small grains from a sample container was necessary. A special spatula made out of stainless steel

304 was prepared to scoop fine grains from samples (Fig. 11c). The margin of the spatula was polished with rough to fine sandpapers not to damage sample grains in scooping samples and curved to maximize the numbers of grains from the bulk sample to other sapphire dishes. The shape of the top of the spatula was determined to maximize grain recovery efficiency through repeated tests using simulant grains.

Remnant magnetizations of samples are one of the essential information for estimating present and past electromagnetic conditions of the asteroid and/or its parent body. To avoid disturbing the remnant magnetization of samples, all handling tools made of stainless steel 304 (such as the nozzles for the tweezer, looped needles, and spatulas) were demagnetized with a commercial demagnetization instrument, HC-31; Hozan Tool Industrial Co., Ltd, Naniwa, Osaka, Japan. After the demagnetization, their sample remnant magnetizations were analyzed in accordance with different distances using a teslameter (Lake Shore Cryotronics Inc. F71), and their magnetizations of zero distances calculated based on fitted curves in plots of distances versus measured magnetizations are as shown in Table 3. The remnant magnetizations of tools should be lower than 0.2 mT, which is in the range of magnetization due to focused ion beam technique applied to samples for magnetization measurement. The remnant magnetization of tools after demagnetization ranged between 0.03 mT and below the detection limit. Only the needle nozzles used for picking up samples were not demagnetized before its use; however, magnetization was analyzed after use and it ranged between 0.9 and 0.2 mT, which is beyond the maximum limit for the magnetization measurement. However, magnetization measurements of Ryugu grains conducted during the initial analysis were between 0.06 and 3.3 mT, which are mostly larger than the remnant magnetizations of the tools. This indicates that Ryugu grains did not experience magnetization with the tools and exhibit relatively high inherent magnetization (Nakamura et al. 2022a, 2022b; Sato et al. 2022).

Sample containers and catcher handling jigs

The touchdown samplings were conducted nominally (Tsuda et al. 2020) and the maximum quantity of samples returned from Ryugu were estimated to be several hundreds of mg (Sawada et al. 2017). Thus, we anticipated that the main target samples for handling would be mm-size, which is significantly larger than sub-mm-sized Itokawa samples from the HY1 mission (Yada et al. 2014). Therefore, developing a new sample container specific for Ryugu samples was required. The instruments used for the initial description of samples impose constraints on the developing process of the new sample container, in

Table 3 Remnant magnetism of nozzles of the vacuum tweezer, looped needles, and spatulas made out of stainless steel 304 analyzed using a teslameter Lake Shore Cryotronics Inc. F71

Date of analysis	Analyzed tools	Quantity	Maximum magnetism (mT)	Note
May 13, 2021	Spatulas	3	0.0253	
	Looped needle	1	N. D	No magnetism
May 20, 2021	Nozzles -S	2	0.0084	No magnetism
	Nozzles -M	1	0.0158	No magnetism
	Nozzles -L	1	0.0328	
June 28, 2021	Nozzles -S03	1	0.0046	No magnetism
July 28, 2021	Nozzles -S	6	0.0283	
	Nozzles -M	4	0.031	
	Nozzles -L	2	0.0155	No magnetism
August 28, 2021	Nozzles—used for Chamber A grains	3	0.2381	
	Nozzles—used for Chamber A grains	3	0.439	
	Nozzles—used for Chamber C grains	4	0.588	
	Nozzles—used for Chamber C grains	2	0.903	
October 12, 2021	Nozzles -S	4	0.0242	
	Spatulas	3	0.004	No magnetism
February 14, 2022	Spatulas	3	0.006	No magnetism
March 16, 2022	Spatulas	3	0.0225	

* N.D.: not detected

particular, the FT-IR and the MO. The sample container should be made out of sapphire glass due to the low absorption of the irradiated light source.

Based on estimated volumes of samples, sample handling tools and initial description methods, the sample container for bulk samples was designed that its maximum size is limited to 23 mm in diameter and 6.5 mm in height, its inside wall should not be vertical but 45° sloped, and that the bottom of its outer case should be open to be transparent to a background of a gold mirror or an infragold plate, which should be set below it (Fig. 12a). A dish-shaped container made of sapphire glass with a diameter of 23 mm fits within an outer case (25 mm in diameter) made of stainless steel 304. The container is sealed with a stainless steel 304 lid and a Viton O-ring is used to hold a quartz glass cover plate. Two types of containers for individual mm-sized grains were prepared, with diameters of 15 and 10 mm. These containers were sealed with a wave washer and a tighten ring by holding the quartz glass cover plate within the stainless-steel outer case (Fig. 12b). Two different cases were also developed to store multiple samples depending on their sizes: 10 and 15 cases for 15- and 10-mm-sized containers. These cases can be stacked on top of each other to store up to several tens of containers safely (Fig. 12c).

The cover plates are required to be removed during the optical observation and the infrared spectrometric analyses, and contamination needs to be avoided during the

observation and the analyses (Fig. 12d). As it is difficult to handle a flat cover quartz glass plate for a sample dish with a pair of normal or even customized tweezers, we prepared a specific attachment for the vacuum tweezer, which was made out of aluminum alloy A6061, to handle and safely move the glass plate between the container and the case (Fig. 12d). Two types of attachments were developed: one for ϕ 23 mm glass plates and another for ϕ 10–15 mm glass plates.

Individual samples have been distributed for initial analyses, phase-2 curation teams, and principal investigators (PIs) of announcement of opportunity (AO) for returned samples. To distribute samples inside the container to researchers without exposing them to air, the Facility-to-Facility Transportation Container (FFTC) was developed by the Phase 2 Kochi Curation team (Fig. 12e, Ito et al. 2020). Its performance test showed that it is capable to seal and preserve samples under positive (+72 kPa) and negative (− 60 kPa) pressure conditions, thus reassuring that the samples are under non-atmospheric condition. Hence, the FFTCs are currently used to distribute samples to the successful PIs following AO submission.

The HY2 sampler catcher was developed for the HY2 mission to store the HY2-returned samples (Sawada et al. 2017). It is 57 mm in length, 48 mm in diameter, and mainly composed of aluminum alloy A6061 (Fig. 13(a)). As previously mentioned, chamber A of the sample

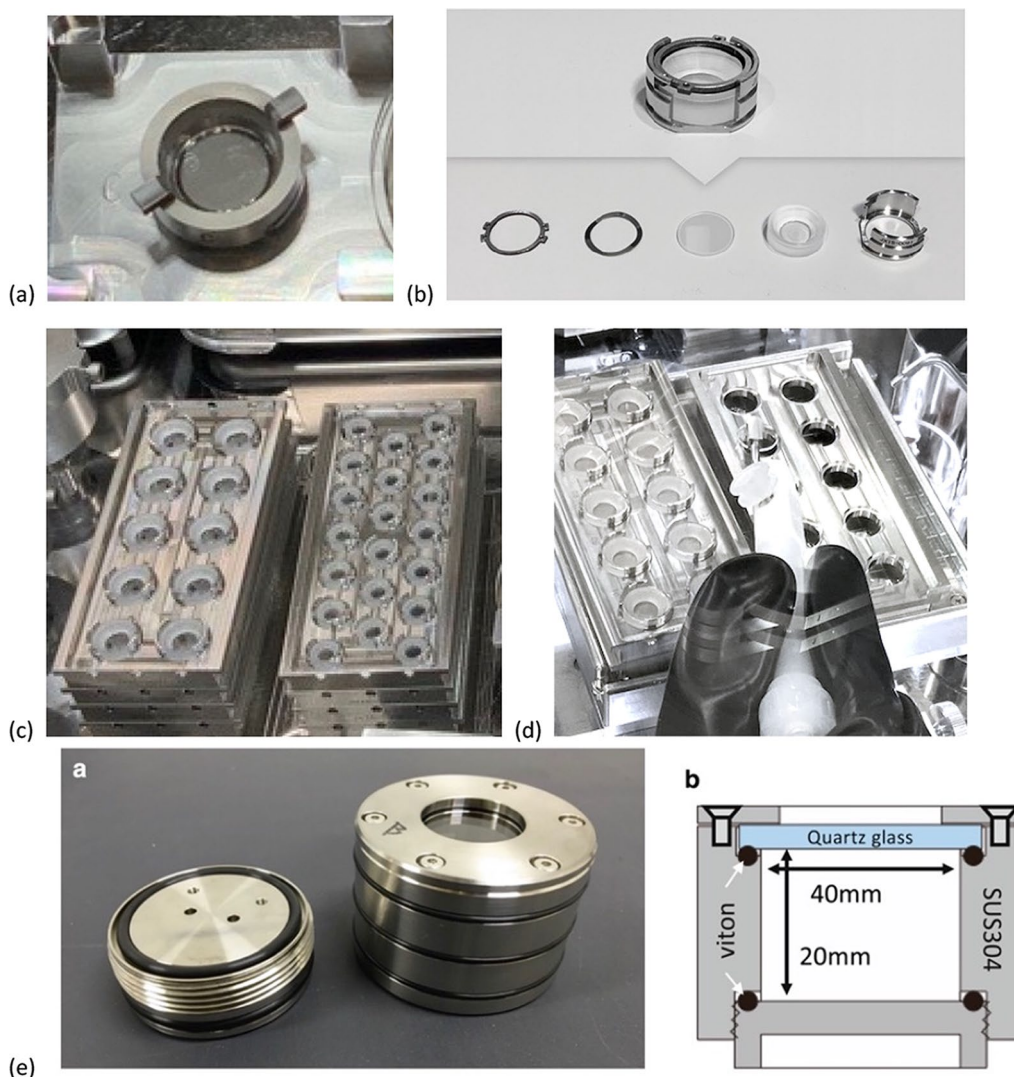


Fig. 12 **a** Image of the sample container for aggregate sample grains. The glass container dish is made of sapphire glass, and its cover plate is made of quartz glass. The container outer diameter is 23 mm. The container and the cover plate are enclosed with a lid and an outer case made of stainless steel 304. **b** A container for an individual sample grain. The outer diameter of the sapphire dish is 15 mm. The outer case of the container is made of stainless steel 304, and the dish as well as the cover plate are tightened with a wave washer in the second left and a leftmost stainless-steel ring. There is also a smaller container whose outer diameter of the sapphire dish is 10 mm. **c** A case for storing multiple containers of diameters 15 mm and 10 mm, respectively. Each case includes 10 and 15 of them, respectively, and can be piled up to store several tens of them. **d** A special nozzle customized to handle a quartz glass cover plate of the container. **e** An FFTC for transportation of individual samples without exposing them to air, from Fig. 1 a and b of Ito et al. (2020)

(See figure on next page.)

Fig. 13 **a** A schematic of the HY2 sampler catcher including returned samples. It is composed of three chambers **A**, **B**, and **C** and a rotation cylinder through which recovered samples are introduced into each chamber. This figure is from Fig. 14 of Sawada et al. (2017) (Fig. 14). **b** A lifting tool (left) and an attachment (right) to be set to the sample catcher. **c** A catcher holding base. **d** The catcher lifting tool (upper) and a catcher rotation tool (lower). **e** A catcher rotation tool to turn upside down the chamber A of the catcher to which a funnel jig is attached. **f** A catcher supporting jig. Screw bolts to connect a cup of the chamber B are situated inside the holes shown in red and pointed by yellow arrows in the viewgraph. **g** A pin to fix a cup to chamber B. The pin is set to a direction pointed by the yellow arrow. **h** A funnel jig to be set to the cup of the chamber B in the direction indicated by the orange arrow. The cup of chamber C is handled in the same manner as shown in **g** and **h**

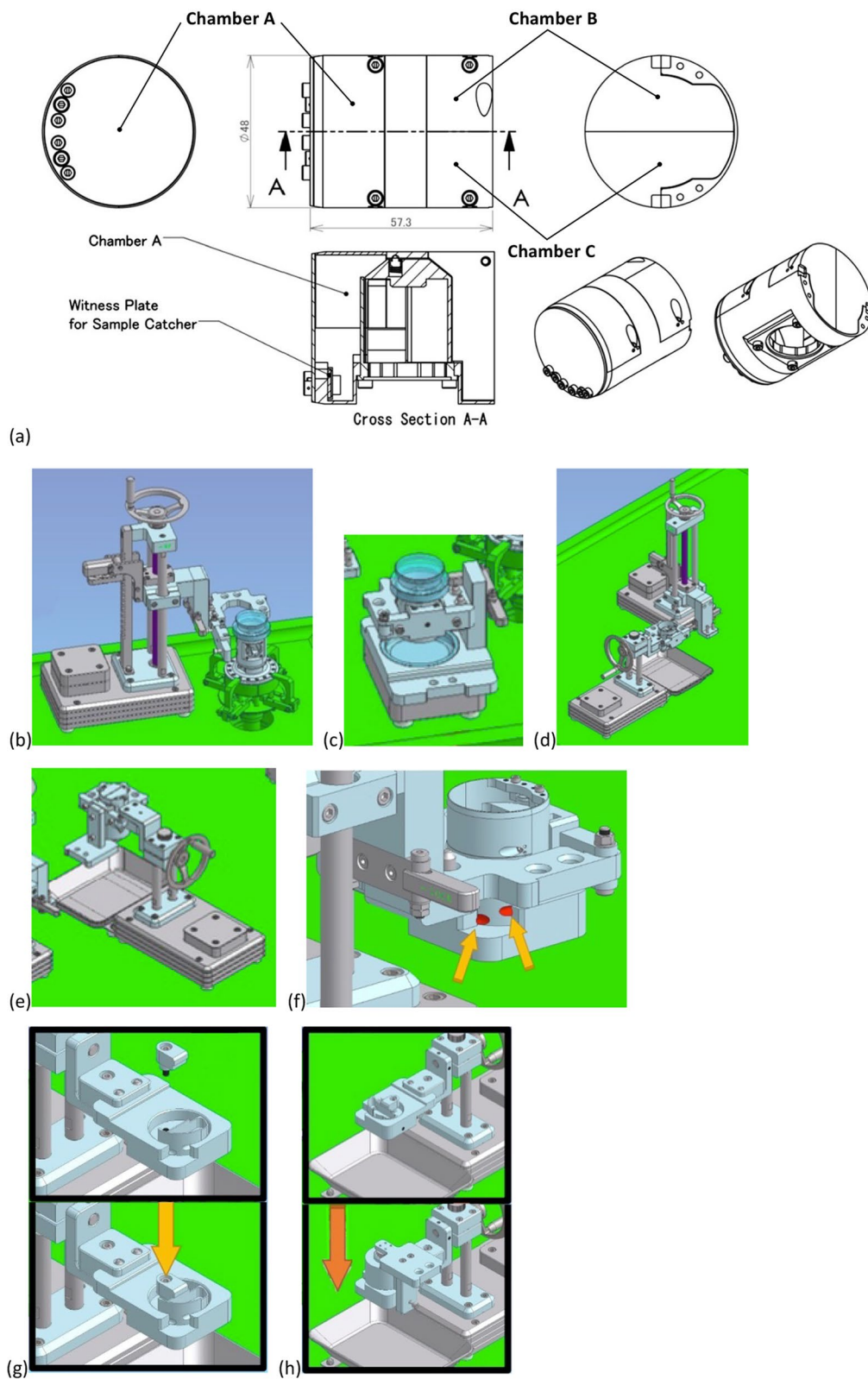


Fig. 13 (See legend on previous page.)

catcher was opened in the vacuumed CC3-2 and the catcher was transported to stage 4 of CC3-3 filled with purified nitrogen to be handled using Viton-coated butyl gloves. Specific tools and jigs were developed to handle the catcher safely and disassemble it to recover the samples.

At first, an attachment made out of aluminum alloy A6061 will be mounted on the sample catcher to allow handling without touching it directly with the gloves in CC3-3 (attachment in darker blue in Fig. 13b). Tools meant to handle the catcher can sustain it with a pair of pins and a pair of holes are present on three sides of the attachment. The attachment set to the sample catcher can cover the opening of the rotation cylinder that contains a certain amount of sample content to avoid sample spilling. The attachment is held with a lifting tool and four screw bolts is removed to detach the catcher from the inner lid of the container (Fig. 13b). The lifting tool consists mainly of stainless steel 304 and allows for moving up and down in 3-mm increments after the removal of the bolts. The catcher will be lifted with the lifting tool and transported to a catcher holding base which is equipped with a pair of pins to sustain the catcher and a quartz glass dish below the catcher (Fig. 13c). The catcher holder base would be transported to the next CC (CC4-1) for further processing. The catcher is transferred to CC4-2 with the catcher holder base, and then the catcher with the attachment is measured for its bulk weight with a balance in CC4-2.

The HY2 sampler catcher with the holder base is sent back to CC4-1 and passed to a catcher rotation tool using the lifting tool (Fig. 13d). A funnel jig, in which a sapphire sample container with a diameter of 23 mm underneath is installed, is set at the opening of chamber A of the catcher. Next, the catcher is rotated upside down and gently hit by a PTFE hammer to let the samples inside fall into the container set in the funnel jig (Fig. 13e). The funnel jig is detached from the catcher and the sapphire container underneath the funnel is removed to recover the samples safely. In the case that samples are plentiful and need to be divided into several containers, samples inside the funnel can be removed into another container with the spatula (scooping tool).

Chambers B and C are different from chamber A in shape and removability from the catcher; the cups-shaped chambers B and C can be detached from the main body of the catcher by removing one or two screw bolts (Fig. 13f). Thus, each of supporting jugs is prepared to sustain and remove chamber B or C and the support jig can be set to the rotation tool to align with either chamber B or C of the catcher sustained by the lifting tool. Next, the screw bolts that connect chamber B or C to

the main body of the catcher is removed to release the cup of chamber B or C into the supporting jig (Fig. 13f). The detached cup of chamber B or C is fixed with a pin and a screw to the supporting jig (Fig. 13g), and entire cup of chamber B or C is covered with the funnel jig with the container for each of the chambers (Fig. 13h). Either chamber B or C with the funnel jigs and the supporting jig is inverted to recover samples from each chamber in the same manner as for chamber A. Notably, all surfaces of the jigs and tools where sample could contact are mirror polished and cleaned following a cleaning protocol for the metal parts to be installed into the CCs (Yoshitake et al. 2021).

Overviews of instruments for initial descriptions

We conducted the initial description process (also known as the phase-1 curation) for HY2-returned samples at the ESCuC using non-destructive methods without exposure to terrestrial air and materials. Five initial instruments were chosen and prepared for the initial description process: an optical microscope, a balance, an FT-IR, an MO, and a visible spectrometer (Miyazaki et al. 2023; Hatakeda et al. 2023; Riu et al. 2022; Yogata et al., in prep; Cho et al. 2022). The optical microscope (Nikon SMZ1270i) was mounted on a moving stage (X, Y, and Z axes mobile) on CC4-2. A modified Mettler-Toledo XP404s balance replacing a standard cover, was placed inside CC4-2. The FT-IR (JASCO VIR-300) was mounted on the FT-IR chamber connected to CC4-2 via the gate valve.

The MO, a hyperspectral imager developed by Institut d'Astrophysique Spatiale (IAS) in France (Pilorget et al. 2022; Riu et al. 2022), was situated on the MO chamber connected to CC3-3 via the gate valve. The optical microscope mounted on CC4-2 was replaced by a digital microscope equipped with six-band filters for visible spectrum during its measurement (Cho et al. 2022). Initial description results for bulk samples were reported by Yada et al. (2022a) and Pilorget et al. (2022). The subsequently obtained initial description results were periodically uploaded to the Ryugu Sample Database System (<https://darts.isas.jaxa.jp/curation/hayabusa2/>) developed by Astromaterials Science Research Group (ASRG), and detailed by Nishimura et al. (2023).

Rehearsal for the HY2 sample return

After the installation of CCs to the HY2 cleanroom in September 2018, the functional tests of CCs were conducted simultaneously with the development of instruments, tools, and jigs. Although the tools for sample processing under vacuum conditions were installed within the CCs (CC3-1, 3-2, and 3-3), the tools, jigs, and containers used under purified nitrogen condition of

Table 4 Rehearsal schedule for Hayabusa2 sample curation

Items	2019												2020												
	Jan	Feb	Mar	Apr	May	Jun	Jul	Aug	Sep	Oct	Nov	Dec	Jan	Feb	Mar	Apr	May	Jun	Jul	Aug	Sep	Oct	Nov	Dec	
Container opening system															Rehearsal			Rehearsal	Rehearsal	Rehearsal					
CC3-1 ~ CC3-3															Rehearsal			Rehearsal	Rehearsal	Rehearsal	Rehearsal	Baking			
CC3-3 ~ CC4-2															Cleanliness test	Baking			Rehearsal	Rehearsal	Rehearsal	Rehearsal	Baking		
Balance															Instrument installation and test measurement				Cable revision				Tare measurement		
Optical microscope			Instrument Installation	Chamber Installation															Image acquisition test	Rehearsal	Image analysis training & test				
FT-IR			Instrument Installation	Chamber Installation															Test measurement	Test & Rehearsal	Rehearsal				
MicrOmega										Operation training in IAS	Chamber Installation								Instrument installation	test & Rehearsal	Rehearsal	Test, calibration, baking			
Visible spectrometer																			meeting & designing				Prototyping & test		

CC3-3, 4-1, and 4-2 were developed first to be used for samples and the catcher. Therefore, approximately 1 year was required following the CC installation to develop and perform a series of procedures for handling the sample catcher and sample simulant. Table 4 shows the rehearsal schedule from 2019 until the HY2 sample return in December 2020. Run-through rehearsal procedures for CC3-1 to CC3-3 under vacuum conditions and for CC3-3 to CC4-2 under purified nitrogen conditions have been performed since March 2020.

Due to the COVID-19 pandemic, the facility was under lockdown in April 2020. After resuming to normal schedule, we performed four run-through rehearsals of processing the catcher from CC3-1 to CC4-2 until November 2020. During the rehearsals, we used grains of synthetic Co-akermanite as simulant in the CCs to allow for clear observation of CC contamination as they are easily identified due to their extraordinary compositions, compared to natural samples (Morioka and Nagasawa 1991). After each rehearsal, the jigs, tools, and/or procedures were assessed for trouble-shooting to improve the sample processing for the actual operation with the HY2-returned samples. In parallel, operations with the balance, optical microscope, FT-IR, MO, and visible spectrometer were assessed in 2020 to be prepared for the initial description procedures for HY2-returned samples.

The CCs were cleaned with clean polyester cloths and isopropyl alcohol as well as ultra-pure water (Millipore, 18.3 MΩ). Next, they were wiped with a dry polyester cloth to remove fine dust on the floor of the CCs after all rehearsals were completed. The CCs were baked at 120–150 °C for more than 3 days, twice under vacuum conditions to evacuate terrestrial gaseous components such as H₂O and O₂ absorbed in the inner surfaces of the CCs. CC3-1, CC3-2, and CC3-3 were maintained under vacuum conditions, while CC4-1 and CC4-2 were purged with purified nitrogen using the circulated nitrogen

purifier for CC4, to recover the condition to the operational level, <−100 °C DP.

Operation for the HY2 returned samples

The HY2 reentry capsule was returned to the Woomera prohibited area (WPA), South Australia, on December 6, 2020 (Yada et al. 2022a; Miura et al. 2022; Nakato et al. 2022). It was immediately recovered and transported to the Quick Look Facility (QLF) in the WPA. Volatile components in the sample container were recovered using the gas extraction and analyses (GAEA) system prepared in the clean booth of the QLF (Miura et al. 2022; Okazaki et al. 2022a). The sample container was sealed under vacuum condition after the extraction of volatile components and transferred to Japan by air.

The sampler container arrived at the ESCuC of JAXA on December 8, 2020 after the successful return of the reentry capsule. The container immediately underwent a series of processes to be disassembled while being maintained under vacuum condition: removal of an ablator attached on the outer lid of the container. As the outer surface of the container was cleaned, the outer lid was first anchored to the container opening system with anchoring jigs to access the inner lid. Next, the inner lid was anchored to the container opening system with rods to remove the outer lid, the frame for the latches, springs, and a non-explosive actuator. Finally, on December 11 (132 h after the reentry capsule landed), the container with the opening system was attached to the bottom of CC3-1, which was designed to keep the Ryugu samples under vacuum conditions and evacuated to high vacuum (~10^{−6} Pa). After the chamber was evacuated, the container was opened on December 14, 2020 using the opening system.

As mentioned previously, the container was opened under static vacuum conditions and the sampler catcher was extracted from the container and transferred to CC3-2. At this point, the sampler container was disassembled



Fig. 14 **a** An image of the bottom of the HY2 sampler container. A certain amount of sample grains are visible. **b** A photograph of the surface of Chamber A of the sample catcher. Fine powdery grains are observed clearly by the illumination with a green LED light, which is useful in recognizing fine dust on the surface of materials. **c** An image of the cover of chamber A of the sample catcher attached on the surface of the electrostatic chuck in CC3-2. The inner surface of the chamber A of the catcher is covered with very fine powdery samples as the reflection on the surface was vague. **d** An image of sample grains in the chamber A of the sample catcher. This is referred from Fig. 1 of Clark (2022). **e** An image of a few sample grains of mm-size, which are preserved in the quartz glass container under the vacuum condition in CC3-2 to date. **f** An image of the powdery samples (pointed by a white arrow) inside the rotation cylinder of the sample catcher situated at the stage 4 in CC3-3. This was shot when the handling jig was about to be attached

into top and bottom pieces. As the catcher was transferred to CC3-2, the container in CC3-1 was observed using the borescope, and a certain number of black-fine particles at the bottom of the sampler container were left behind in CC3-1 (Fig. 14a). The gate valve between CC3-1 and CC3-2 was closed after the catcher transfer and CC3-1 has been maintained under vacuum conditions until the present date.

In CC3-2, the catcher was first transported to stage 3 (for layout of each stage, see Fig. 5a) and the surface of the cover of chamber A of the catcher was cleaned with a PTFE spatula. The outer surface of chamber A appeared to be covered with fine powder materials (Fig. 14b), which were removed following the cleaning process (PTFE spatula cleaning). Next, the catcher was transported to stage 1 to remove four screw bolts that were used to fix the cover of the catcher to the catcher body. The cover was removed with an electrostatic chuck (Fig. 14c). After removing the cover, samples inside chamber A of the catcher were first observed with the naked eye (Fig. 14d). The catcher was subsequently transported to stage 2 to observe with a borescope, and a few sample grains of mm-size were picked up using the sample pickup tool and stored in the quartz glass container (Fig. 14e). Next the catcher was transported to stage 3, and a quartz glass cover was placed over the opening of chamber A. Finally, the catcher was transported to CC3-3 on December 16, 2020, and the samples inside CC3-2 have been stored under vacuum conditions to date (the gate valve between CC3-2 and CC3-3 was closed).

CC3-3 including the catcher was purged with purified nitrogen slowly for 5 h at atmospheric pressure. Next, the circulation of CC3-3 purified nitrogen was initiated to maintain high purification levels as well as under -100 °C DP. When the handling jig was attached to the catcher at stage 4, a small amount of powder sample was observed inside the rotation cylinder of the catcher (Fig. 14f). These samples remained inside the rotation cylinder as the handling jig was attached, which we plan to recover in the near future.

As the handling jig was fixed to the catcher, four screw bolts that connect the catcher to the inner lid were removed with a hex key, which allowed for the detachment of the catcher from the inner lid using the lifting tool. The catcher was attached to the catcher holding base and transferred to CC4-1. Subsequently, the catcher with the holding base was transported to CC4-2 with the lifting tool in CC4-1 and was observed under the optical microscope as well as weighted using the balance. The total weight of the returned samples inside the catcher was approximately 5.42 g (Yada et al. 2022a).

Next, the catcher was transferred back to CC4-1 to be disassembled using the tools and jigs mentioned before, and to recover samples from each of its chambers. The images of bulk samples recovered from chambers A and C in the sapphire dishes are shown in the appendix of Fig. 2 in the study by Yada et al. (2022a); 3 sample dishes from chamber A, 1 sample dish from chamber B, and 3 sample dishes, as well as 15 individual large grains, were recovered from chamber C. Samples were analyzed using the FT-IR, MO, and visible spectrometer (for details see Yada et al. 2022a and Pilorget et al. 2022). After the initial descriptions of bulk samples, individual grains were picked up using the vacuum tweezers and transferred to each container to be described in the same manner as the bulk samples.

Concluding remarks

The initial description of individual sample grains is still ongoing (May 2023). All tools, jigs, containers, and instruments developed for the HY2-returned samples are fully operational and assist in the successful sample curation within the ESCuC. The successful curation activities for the HY2-returned samples allowed for significant scientific discoveries following initial analysis and phase 2 curation teams (Nakamura et al. 2022a, 2022b; Yokoyama et al. 2022; Ito et al. 2022; Okazaki et al. 2022b; Noguchi et al. 2022; Naraoka et al. 2023; Yabuta et al. 2023). The developed techniques and knowledge acquired in the field of extraterrestrial material curation at JAXA is invaluable for curatorial projects of other future sample-return missions such as the Origins, Spectral Interpretation, Resource Identification, Security-Regolith Explorer (OSIRIS-REx) (Lauretta et al. 2019), and Martian Moon eXploration (MMX) (Usui et al. 2020).

Abbreviations

ADC	Aluminum die-cast alloy
AO	Announcement of opportunity
API-MS	Atmospheric pressure ionization mass spectrometer
ASRG	Astromaterials Science Research Group
BA	Bright annealing
C-type	Carbonaceous-type
CC	Clean chamber
DP	Dew point
DCM	Dichloromethane
EP	Electro polishing
EPS	Electronics and pipes shaft
ESCuC	Extraterrestrial sample curation center
FT-IR	Fourier transform infrared spectrometer
GAEA	Gas extraction and analyses system
GC-MS	Gas chromatograph-mass spectroscopy
HY1	Hayabusa1
HY2	Hayabusa2
IAS	Institut d'Astrophysique Spatiale
ISAS	Institute of Space and Astronautical Sciences
ISO	International Organization for Standardization
JAXA	Japan Aerospace Exploration Agency
MMX	Martian Moons Exploration

MO	MicrOmega
OSIRIS-REX	Origins, Spectral Interpretation, Resource Identification, Security-Regolith Explorer
PBS	Phosphate buffered saline
PE	Polyethylene
PEEK	Polyether ether ketone
PFA	Perfluoroalkoxy alkane
PI	Principal investigator
PP	Polypropylene
PS	Pipes shaft
PTFE	Polytetrafluoroethylene
PVC	Polyvinyl chloride
QLF	Quick look facility
RGA-QMS	Residual gas analyzer quadrupole mass spectrometer
RS	Return shaft
S-type	Stony-type
TD-GC-MS	Thermal decomposition gas chromatograph-mass spectroscopy
ULPA	Ultra low particulate air filters
VPD-ICP-MS	Vapor phase decomposition inductively coupled plasma mass spectroscopy
WPA	Woomera Prohibited Area

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Author contributions

TY, MA, MN, ST, HY, TO, HS, RO, YT, YNM, MI, MU, YK, AN, KS, KU, NT, NS, AY, HN, and NI discussed specifications and procedures for the HY2-returned sample curation. TY, MA, MN, HS, RO, YT, KS, TO, AN, MY, YN, KY, AM, SF, ASI, SN, KH, KK, KU, MU, and HN conducted tests and developed sample containers, sample processing jigs, and tools for the curation of the HY2-returned samples. YK, AN, YH, KK, MY, and SS conducted environment tests for the cleanrooms and CCs. YY, MI, AN, KY, and AM conducted magnetization and demagnetization measurements for tools used for handling the HY2-returned samples. TY, MN, YH, MY, AN, KY, AM, YY, KK, and SS summarized and managed the data for manuscript preparation. MA, TO, ST, HY, and TU supervised the curatorial activities. All authors read and approved the final manuscript.

Data availability

All figures and tables of this report are available in https://data.darts.isas.jaxa.jp/pub/curation/paper/Yada_2023/.

Declarations

Ethics approval and consent to participate

Not applicable.

Consent for publication

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Competing interests

The authors declare that they have no competing interests.

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