ISSN 2433-2232(Online)
JAXA-SP-22-006E

JAXA Special Publication

Contamination analyses of clean rooms and clean chambers at the Extraterrestrial Sample Curation Center of JAXA in 2021 and 2022

February 2023

Japan Aerospace Exploration Agency

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ABSTRACT

The Extraterrestrial Sample Curation Center of JAXA receives samples returned by asteroidal explorer. Such returned samples are handled by meticulously cleaned tools in the nitrogen-circulation-type clean chamber to prevent contamination from earth materials. In May 2022, two new glove boxes (GB6 and GB7) were installed to our curation facility for the storage of samples returned from initial analyses of Hayabusa2, and general sample handling in high-purity nitrogen respectively. These glove boxes were confirmed to have the same level of cleanliness as GB1 from the results of the contamination analyses. New ISO Class 6 clean room (ISO-14644-1), clean chambers and glove boxes have also been designed for receiving the samples from the OSIRIS-REx mission returning to Earth in 2023.

Human activity, tools and construction work are the risk to introduce the contaminants into the clean room and clean chambers. To ensure that extraterrestrial samples are maintained in a clean environment, it is essential to continuously monitor the cleanliness of clean rooms, clean chambers, and glove boxes in which samples are processed and stored. Thus, in this study, we employ results of annual contamination analyses to assess the cleanliness level of the environment where the samples were processed and stored based on ISO standards (ISO-14644-8, ISO-14644-10), and it was confirmed that the cleanliness level is kept until 2022 even after the Ryugu samples were returned and processed. Cultivation-based microbial contamination analysis of both clean rooms and clean chambers detected no microorganisms. In February 2022, comprehensive two-dimensional gas chromatography Time-Of-Flight Mass Spectrometer was introduced in our group, which enabled analysis of organic substances in our laboratory. It has become possible to obtain organic materials data that interpolate environmental evaluation that are outsourced to outside contractors, which can only be performed once or twice a year. The results of GC analysis indicated the increase of organics in the adjacent clean room after starting the new clean room construction.

1 INTRODUCTION

The Extraterrestrial Sample Curation Center of JAXA (ESCuC) has been established to store and handle the extraterrestrial samples recovered in the sample return missions such as Hayabusa, and Hayabusa2[1-3]. Those samples are significantly valuable as they are directly brought back from the space to the earth without any exposure to terrestrial atmosphere and considered to preserve their original characteristics. Therefore, it is essential to prevent the samples from contamination by the earth's materials and atmosphere during the sample processing and storage.

The ESCuC houses eight clean rooms (seven as ISO-14644-1 Class 6 and one as ISO-14644-1 Class 7) as of fall 2022. Three of ISO Class 6 (Planetary Sample Handling Room [PSHR] 3, Sample Preparation Room [SPR] 2, and SPR3) have been newly constructed in 2022 (Fig. 1). Purified nitrogen circulating and vacuum clean chambers were installed to store and process samples from S-type near-Earth asteroid 25143 Itokawa (CC1 and CC2) and from C-type near-Earth asteroid 162173 Ryugu (CC3 and CC4) in PSHR 1 and 2, respectively (both rooms are ISO Class 6). Installation of a new clean chamber in PSHR3 is planned during 2023 to receive samples from B-type asteroid 101955 Bennu by NASA's OSIRIS-REx mission. In the ESCuC, we dedicate the clean chambers to the initial descriptions and pristine samples processing prior to distribution whereas we use the glove boxes to prepare the samples for analyses and store them that have undergone the various analyses. Three purified nitrogen circulating glove boxes (GB2, GB6, and GB7) are present in Electron Microscope Room (EMR, ISO Class 6). GB2 and GB6 are used to store previously allocated samples, most of which were processed for various examinations and returned to the ESCuC. Nitrogen gas replacement type glove boxes are also setup in the clean rooms: two (GB1, and GB5) in EMR and two (GB3, and GB4) in Manufacturing and Cleaning Room (MCR, ISO Class 7). They are used for sample processing, microscopic observation, and/or IR spectrometric analysis.

Cleanliness of all the glove boxes and clean chambers are monitored and assessed in two ways on a real-time basis. All the glove boxes and clean chambers are equipped with dew-point hydrometers to monitor the moisture content. Oxygen sensors are additionally installed to the purified nitrogen circulating glove boxes to monitor the oxygen content whereas the clean chambers are connected to Atmospheric Pressure Ionization Mass Spectrometer (API-MS) (for Hayabusa : UG-510, Renesas east japan semiconductor Co. Ltd., for Hayabusa2 : API-200, NIPPON API Co Ltd.) for monitoring the amount of gases including H_2O, O_2, CO_2, and CH_4. Independent from the real-time base monitoring, the analyses of the organic and inorganic contaminants in each clean room and clean chamber in the ESCuC have been conducted annually by the outsourcing company (Renesas Semiconductor Manufacturing Co., Ltd.) since 2013. Yoshitake and colleagues[4] evaluated the results from 2013 to 2020 with the information of the cleaning procedures of the handling tools. This study revealed that the clean rooms and clean chambers have remained clean since construction, providing the foundation of the cleanliness of the environment and cleaning protocol at this facility. However, an annual contamination analyses is still limited to evaluate the variation of contaminants and their concentrations throughout the year, and analyze them in timely manner when possible environmental change in the clean rooms and/or clean chambers occurs by maintenance, construction or accident. This highlights the necessity of frequent contamination analyses to more accurately assess the cleanliness level of the environment of the clean rooms and clean chambers that house the pristine extraterrestrial samples.

Here, we present the results of organic and inorganic contamination analyses of the clean rooms and clean chambers from 2021 to 2022 based on the data from the outsourcing company, and compare these with the data from 2018 to 2020 by Yoshitake and colleagues[4] to evaluate the cleanliness level of the environment over the years in our facility. In particular, we focus on before and after the events that may have introduced the contaminants, such as construction of the clean rooms in 2022, and receiving the extraterrestrial samples in December 2020, and whether any changes of the cleanliness level have occurred across the events. We also present brief results of first in-house organic analysis conducted in this facility, using newly installed Comprehensive Two-Dimensional Gas Chromatography Time-of-Flight Mass Spectrometer (GC×GC-TOFMS), to close the gap of the time frame of the routine annual inorganic and organic contamination analyses. Lastly, we briefly discuss the cleanliness level of the clean rooms and clean chambers based on the results derived from the microbial contamination analysis conducted by our team in December 2021.

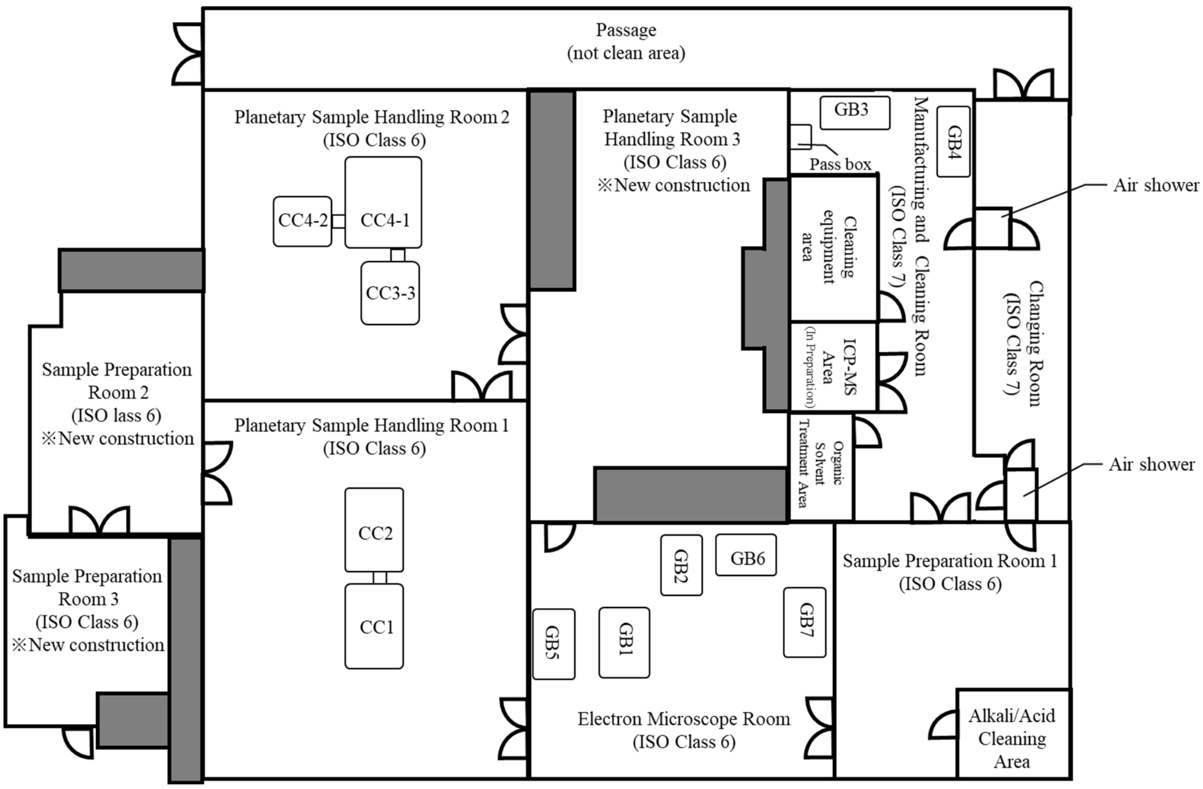

Fig. 1 Clean room layout in the ESCuC

2 METHODS

2.1 Annual organic and inorganic contamination analyses

The annual organic and inorganic contamination analyses have been conducted on both Hayabusa and Hayabusa2 samples collected from four clean rooms (PSHR1, PSHR2, EMR [ISO Class 6], and MCR [ISO Class 7]) and clean chambers (CC2, CC3-3, CC4-1, CC4-2) since 2013 by the outsourcing company. The glove boxes (GB1, GB6, GB7) and desiccators were also analyzed if necessary. Organics, dissolved inorganic ions, and metal elements were sampled and analyzed by solid-phase adsorption, wafer exposure, and impinger methods. We received the raw data from the outsourcing company and transformed the data into the graph presented in this study. As Yoshitake and colleagues presented the results of the contamination analyses of each year from 2013 to 2020[4], we only present the averaged data spanning eight years in this study; however, organic analyses were conducted only using the data from 2018 to 2022 as the analysis method changed from 2018.

2.1.1 Solid-phase adsorption method

The organic matter was obtained at a rate of 0.5 L/min for 1.5 h using a two adsorption tubes (adsorbent: Tenax-GR) and two metering pumps. After sampling, the adsorption tubes were returned to the outsourcing company in a special sealed case. Organic contaminants on the adsorption tubes were extracted with the thermal desorption system (GERSTEL TDS A2) heated up to 280°C, concentrated with a cold trap at -100°C, and analyzed using a gas chromatographic mass spectrometer (GC-MS; Agilent 6890NGC/5973MSD). Concentrations of organic components were calculated semi-quantitatively using peak area of standard reagents (eicosane).

2.1.2 Wafer exposure method

To estimate the amount of contamination caused by metal elements and organic substances on the surface of the clean chambers and clean rooms, the contaminants were collected using the wafer exposure method. Silicon wafer in 8-inch diameter was exposed in the clean rooms and clean chambers for approximately 15–20 h. After sampling, the wafers were returned to the outsourcing company in a dedicated sealed case. For measuring the organic materials, the organic impurities attached to the wafer were removed using a temperature-raising degassing device heated up to 400°C. Extracted organics were recaptured in adsorption tubes (adsorbent: Tenax-GR) and then analyzed using GC-MS (Agilent 6890NGC/5973MSD) equipped with the thermal desorption system (GERSTEL TDS A2). For measuring the inorganic materials, the samples on the wafers were decomposed in the gas phase with 50% hydrofluoric acid for 10 minutes, and then metal impurities were recovered by droplet scanning with mixed solution of 2% hydrofluoric acid and 2% hydrogen peroxide. The recovered liquid was measured by ICP-MS (Thermo Fisher Scientific ELEMENT2). When performing the wafer exposure method in a clean chamber, it is necessary to move the extraterrestrial samples to another clean chamber to avoid a contamination from wafer to samples. Analyses on the samples from CC3-3 was not carried out after receiving Ryugu samples because CC3-3 is used as a temporary storage place of the samples when put the water into the clean chamber.

2.1.3 Impinger method

To estimate the contamination of floating materials (inorganic and metal ions) in atmosphere in the clean rooms, an impinger method was used to obtain ambient air at a flow rate of 1.0 L/min for 17–19 h. After sampling, the sample solutions were transferred to a storage container and returned to outsourcing company. The sample solutions were measured using two instruments: a double-focusing inductively coupled plasma-mass spectrometer (ICP-MS; Thermo Fisher Scientific ELEMENT2) for metal ions, ion chromatography (IC; Dionex DX-500 and DX-120) for inorganic ions (anions and cations).

2.2 In-house organic contamination analysis

Organic materials for in-house contamination analysis were sampled by solid-phase adsorption method. Air samples in the clean rooms were collected with an adsorption tube (adsorbent: Tenax-TA and activated carbon) and metering pump at a rate of 0.5 L/min for 0.5 h. Organics were then measured by Comprehensive Two-Dimensional Gas Chromatography Time-of-Flight mass spectrometer (GC×GC-TOFMS; Agilent 8890GC / LECO Pegasus BT4D) with the thermal desorption system (GL Sciences TD265). We chose GC×GC-TOFMS as this spectrometer significantly improves the qualitative capability by connecting two columns with different phase selectivity and using modulator. Organic materials collected on the adsorption tube were extracted by a thermal desorption device heated up to 280°C, and introduced to GC.

2.3 Microbial contamination analysis

Samples for microbial contamination analysis were collected from three locations in PSHR2: floor, floor inside of the CC4-1, and inner surface of the glove of the CC4-1 on December 9th 2021. Positive control (P.C.) sample was also collected from the floor in the laboratory outside the clean room. Sampling areas of 300 cm^2 were swept using polyurethane foam swabs, and swept was repeated three times with different directions. Swept swab was soaked in 15 mL Phosphate-buffered saline (PBS) solution in sterilized plastic tubes, and stirred using a Vortex Mixer for 10 seconds. Sample solutions were placed on culture media and cultivated with different conditions (Table 1). Sterilized PBS solution was placed on a culture medium, which was used as a negative control (N.C.) sample. After the cultivation, the numbers of colonies were counted.

Table 1 Types of media and culture conditions in microbial contamination analysis

Culture media	**Sample amount (ml)**	**Number of media**	**Culture condition**
TSA plate	0.1	4	2 days, 35ºC
Blood agar plate	0.1	2	2 days, 37 ºC
R2A plate	0.1	2	7 days, 25 ºC
Saboraud-dextrose plate	0.4	1	7 days, 30 ºC
Saboraud-dextrose with chloramphenicol plate	0.4	2	7 days, 30 ºC
Poteto-dextrose plate	0.4	1	7 days, 30 ºC
Thioglycolate plate	1.0	2	7 days, Room temperature, Anaerobic

3 RESULTS AND DISCUSSION

3.1 Annual contamination analyses

The Hayabusa2 space craft brought the samples from C-type near earth asteroid 162173 Ryugu to the earth on 6th of December in 2020. Since the samples were transported to the ESCuC, they have been processed, analyzed and stored in the clean chambers (CC3 and CC4) in PSHR2. Thus, the environment especially in PSHR2 and clean chambers of CC3-3, CC4-1, and CC4-2 may have been changed after receiving the Ryugu returned samples. Therefore, we compared the annual contamination analyses data focusing on data between before introducing the returned samples to the environment and after that.

3.1.1 Solid-phase adsorption method

Organic materials in the clean rooms were collected by solid-phase adsorption method and analyzed by GC-MS. Total organic compounds in 2021 are 4~9×10^3 ng/m^3 in all clean rooms, which are almost the same amounts as those before the sample return or even lower (Fig. 2). This demonstrates that no contaminants were introduced into the clean rooms by the Ryugu sample return. The similar concentrations of the organics found between the MCR (ISO Class7 clean room) and other three clean rooms of ISO Class 6 (PSHR, PSHR2 and EMR) indicate that the MCR is kept as the same cleanliness level as the other ISO Class6 clean rooms (Fig. 2). For individual components, toluene and cyclosiloxane mainly derived from building materials, and benzene and 2-ethyl-1-hexanol derived from outside air were predominantly detected.

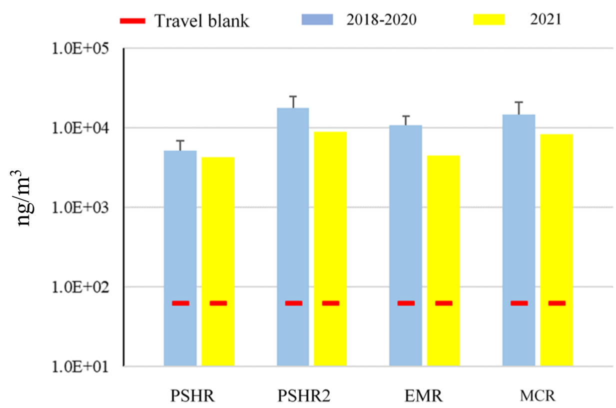

Fig. 2 Total organic compounds in the air determined using GC-MS by the solid-phase adsorption method

3.1.2 Wafer exposure method

Organic and inorganic particles fallen on the exposed silicon wafers in clean rooms, clean chambers and three glove boxes (GB1, GB6, and GB7) were analyzed. Total organic compounds in the clean rooms did not significantly change between before and after the sample return (1 to 10 ng/cm^2, Fig. 3), which is consistent with the result by the solid-phase adsorption method (Fig. 3). Organics are always much less present in the clean chambers than those in the clean rooms and glove boxes (0.2 to 0.7 ng/cm^2), and those in CC4-1 and CC4-2 for Hayabusa2 appear to decrease after the sample return. These decreased amounts of organics might be resulted from the refurbishment and baking of the chambers (CC4-1 and CC4-2) in November 2020 immediately before the sample return. Concentrations of the organics in the glove boxes (GB1, GB6 and GB7) are almost the same level as the clean rooms (PSHR, PSHR2, EMR and MCR). Those in the newly installed GB6 and GB7 in March 2022 are less than in GB1. This may be explained by two factors: the new glove boxes have never been used before the analyses, and are equipped with the circulation type nitrogen purifier.

Overall, the results of the metallic elements indicate that most of the elements have similar concentrations or even less relative to the blank data (Fig. 4a-4j). Comparisons of the concentrations of the metallic elements between before and after the sample return do not show a significant difference with only few exceptions, suggesting no contaminants are present. Those few exceptions include aluminum in PSHR2 (Fig. 4b), potassium and sodium in EMR (Fig. 4c) and barium in CC4-1 (Fig. 4g), and all of their concentrations increased in 2021. Aluminum foil is commonly used in PSHR for various purposes, and considered to be the possible contaminant. Increased concentration of potassium in EMR might be caused by human activity because potassium is one of the common contaminants derived from a living body. Similarly, concentration of sodium, another common contaminant derived from humans, was also increased in 2021 along with potassium though the sodium concentration is below the blank level. Barium was often detected as highly concentrated in PSHR and EMR before 2020 (Fig. 4a and 4c). Barium contamination might be caused by rubber, plastics and printing ink used in clean rooms because barium is also used as an additive in these materials.

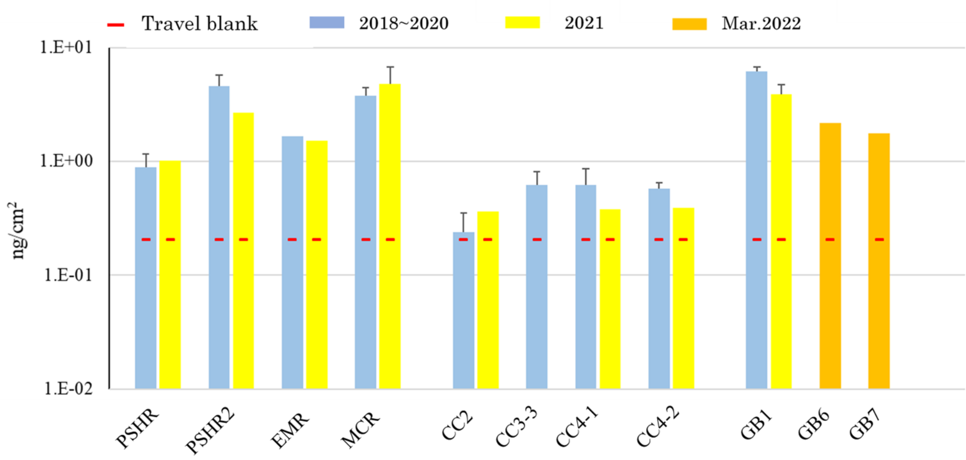

Fig. 3 Total organic compounds on the surface of the wafers determined using GC-MS by the wafer exposure method

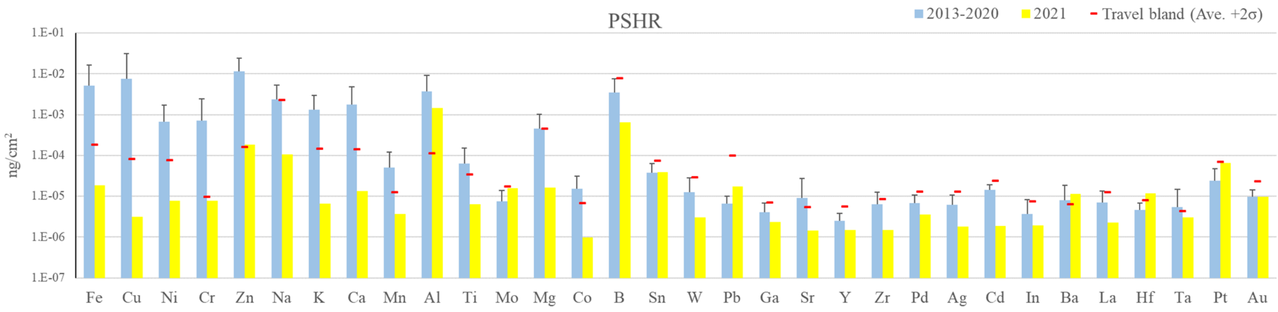

Fig. 4a Metallic elements on the surface of wafer in the PSHR determined using ICP-MS by wafer exposure method

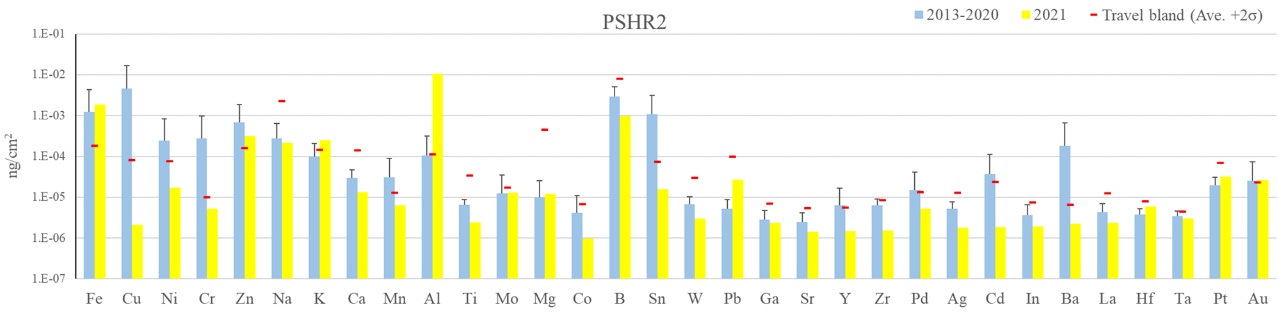

Fig. 4b Metallic elements on the surface of wafer in the PSHR2 determined using ICP-MS by wafer exposure method

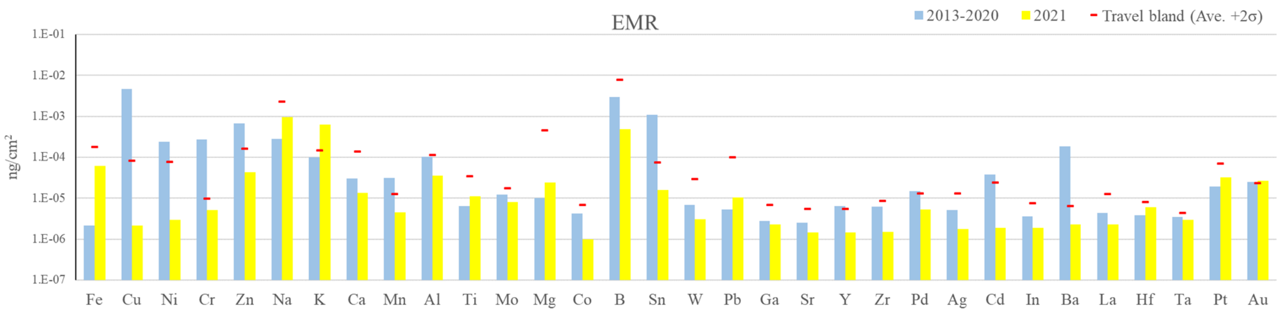

Fig. 4c Metallic elements on the surface of wafer in the EMR determined using ICP-MS by wafer exposure method

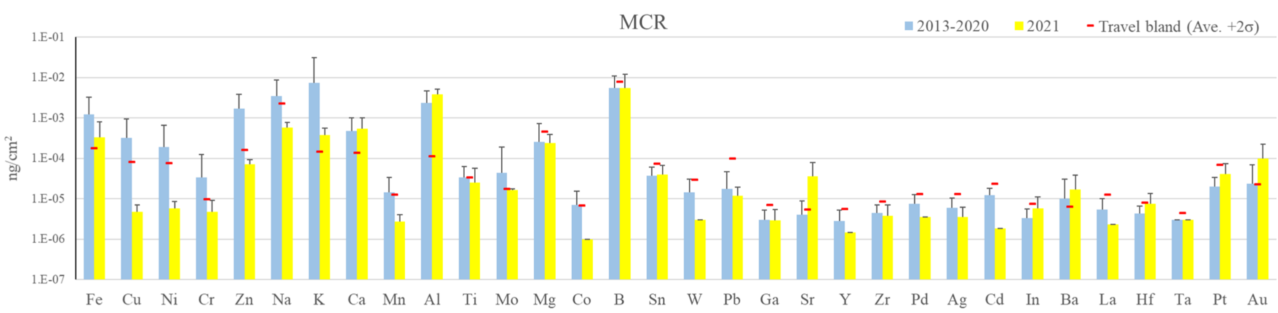

Fig. 4d Metallic elements on the surface of wafer in the MCR determined using ICP-MS by wafer exposure method

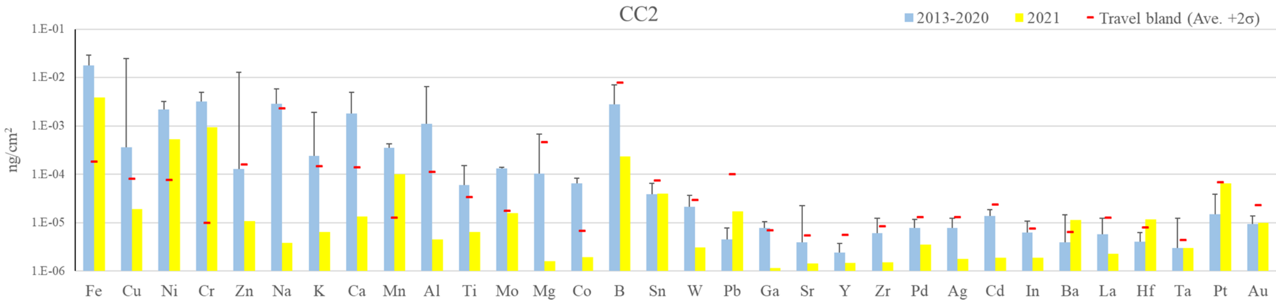

Fig. 4e Metallic elements on the surface of wafer in the CC2 determined using ICP-MS by wafer exposure method

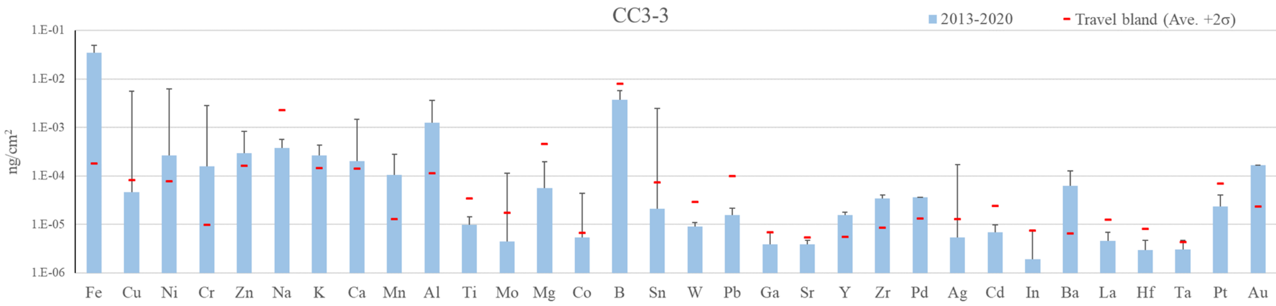

Fig. 4f Metallic elements on the surface of wafer in the CC3-3 determined using ICP-MS by wafer exposure method

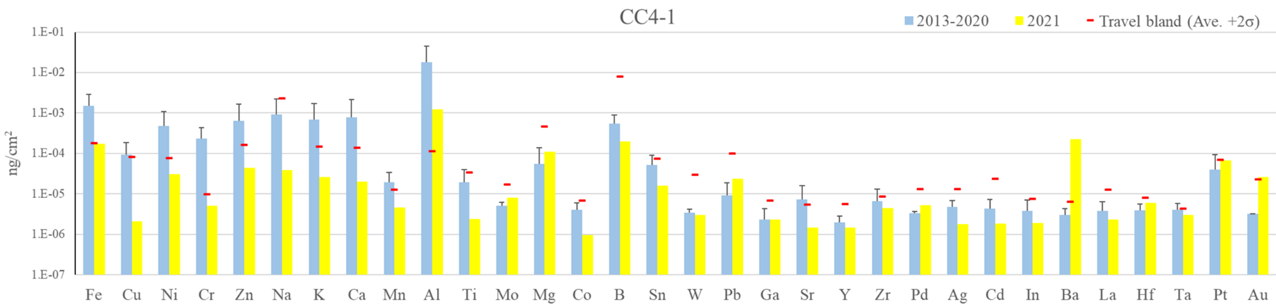

Fig. 4g Metallic elements on the surface of wafer in the CC4-1 determined using ICP-MS by wafer exposure method

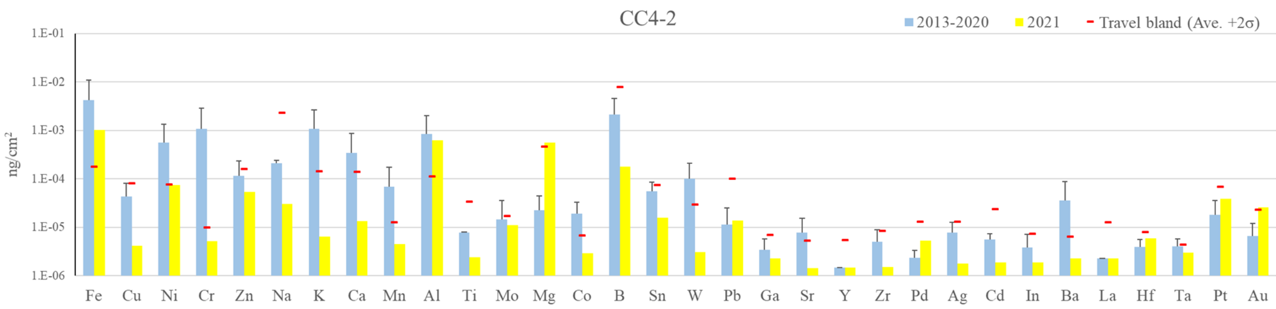

Fig. 4h Metallic elements on the surface of wafer in the CC4-2 determined using ICP-MS by wafer exposure method

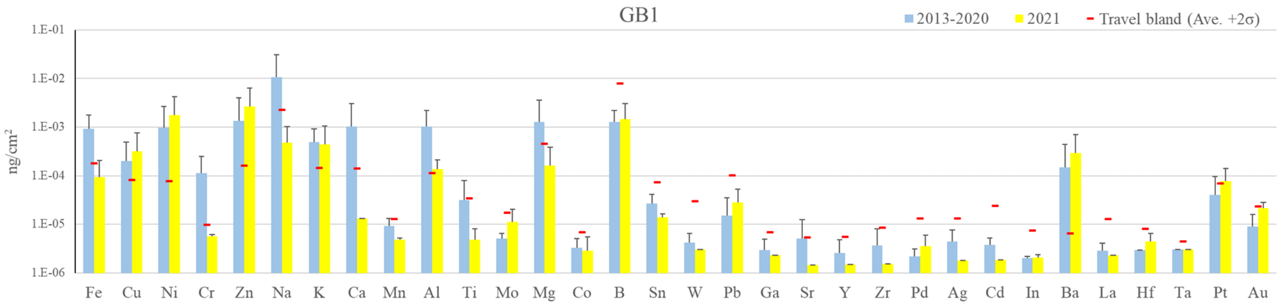

Fig. 4i Metallic elements on the surface of wafer in the GB1 determined using ICP-MS by wafer exposure method

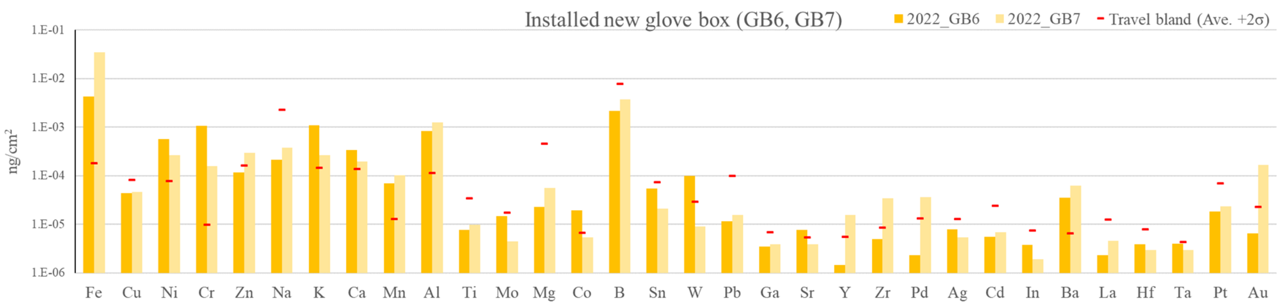

Fig. 4j Metallic elements on the surface of wafer in the GB6 and GB7 determined using ICP-MS by wafer exposure method

3.1.3 Impinger method

Samples collected by the impinger method were analyzed using ion chromatography and ICP-MS. Ion chromatographic data in all the clean rooms indicate no significant difference of the environment between before and after the Ryugu sample return, confirming no contaminants were introduced into the clean rooms (Fig. 5a-5d). Relatively high concentrations (100 to 1000 ng/m^3) of acetic acid, formic acid, NH_4^+, and NO_2^- and NO_3^- are considered to be derived from building materials, and human activities, and may also be derived from ambient air outside the clean rooms (4).

Most of metallic elements analyzed using ICP-MS are lower than results of wafer exposure method and below the blank level (Fig. 6a-6d). This is because the wafer exposure method uses an acid solution whereas the impinger method uses ultrapure water as a solvent, so the metallic elements is considered to be dissolved sparingly in solvent. In contrast, only the concentrations of boron, sodium and potassium in PSHR2 are significantly higher than their respective blank samples and especially the two latter increased after the sample return. Curation activities in PSHR2 have been significantly increased due to sample processing and analyses of Ryugu returned samples after December 2020 compared to the other clean rooms. This suggests that the increased concentrations of sodium and potassium in PSHR2 may be associated with the increase of human activity.

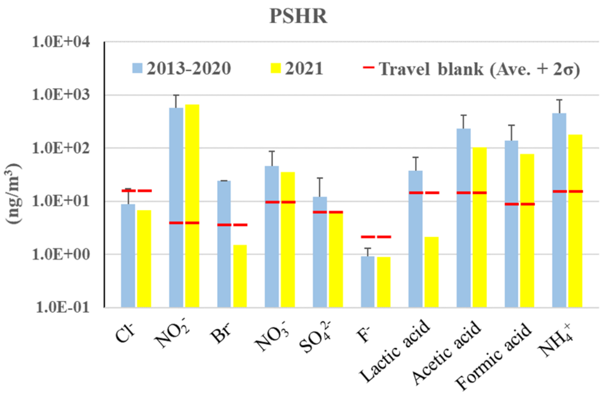

Fig. 5a Inorganic ions in PSHR determined using IC by impinger method

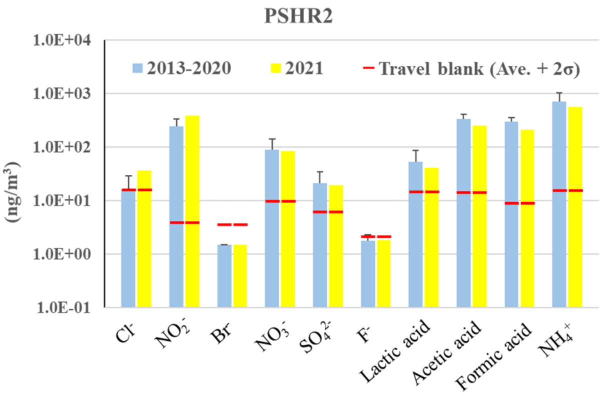

Fig. 5b Inorganic ions in PSHR2 determined using IC by impinger method

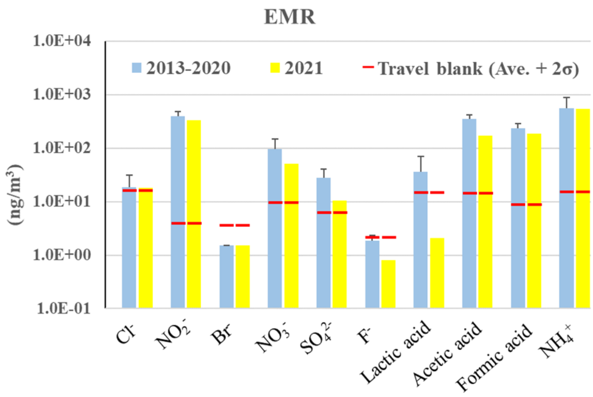

Fig. 5c Inorganic ions in EMR determined using IC by impinger method

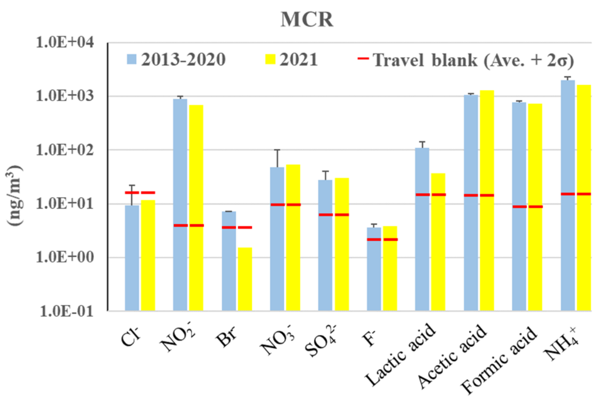

Fig. 5d Inorganic ions in MCR determined using IC by impinger method

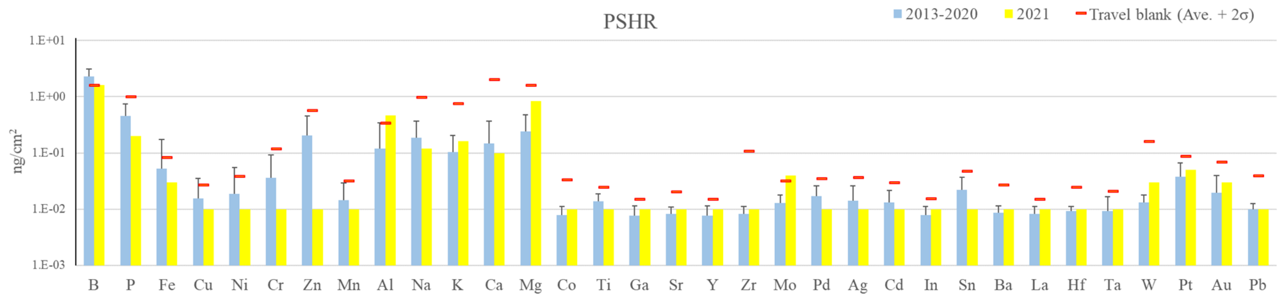

Fig. 6a Dissolved metallic elements in PSHR determined using IC by impinger method

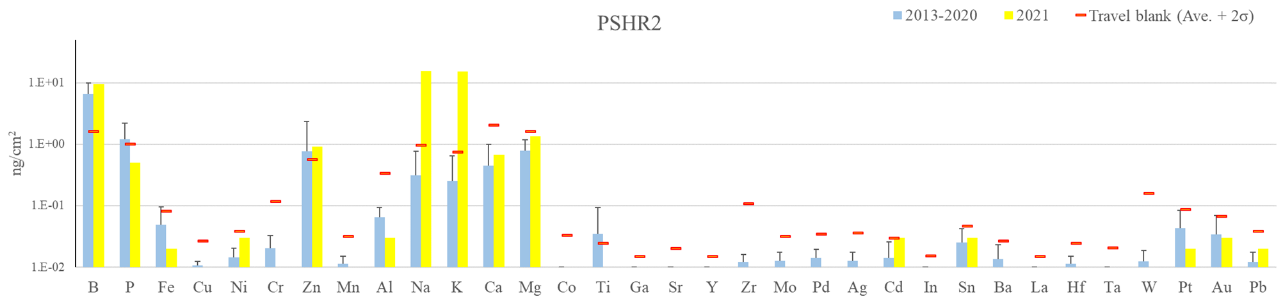

Fig. 6b Dissolved metallic elements in PSHR2 determined using IC by impinger method

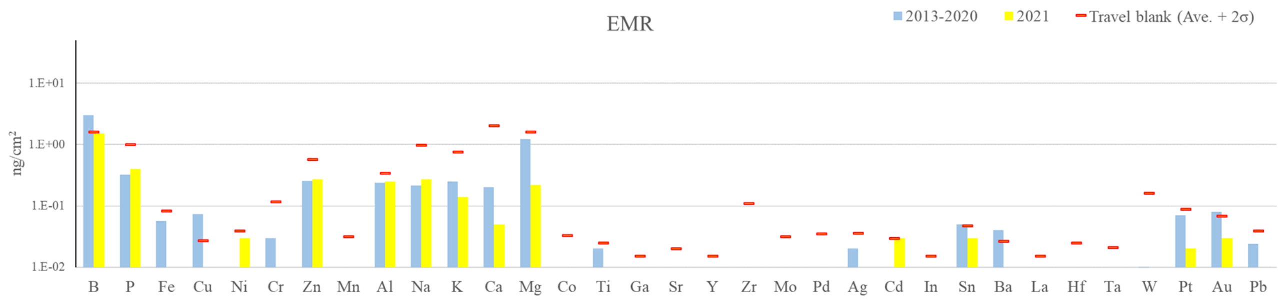

Fig. 6c Dissolved metallic elements in EMR determined using IC by impinger method

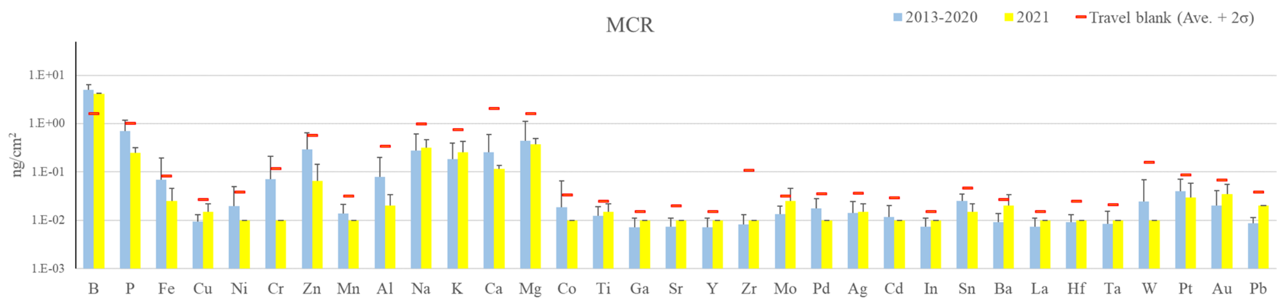

Fig. 6d Dissolved metallic elements in MCR determined using IC by impinger method

3.1.4 Cleanliness of the clean rooms and clean chambers in ISO standard

Based on the results of the annual environmental assessment, the cleanliness of the clean room was classified according to ISO standards. Table 2 shows the results of the solid-phase adsorption and impinger methods according to ISO-14644-8 indicating the air cleanliness by chemical concentration (ACC). Table 3 shows the results of the wafer exposure method according to ISO-14644-10 indicating the surface cleanliness by chemical contamination (SCC).

Table 2 Cleanliness classification of air cleanliness by chemical concentration for each location (ISO-14644-8)

Location	**Air cleanliness by chemical concentration (ACC)**		
	Total organic matter	**Metallic elements*1**	**Inorganic ions*1**
Planetary Sample Handling Rm. 1 (PSHR)	ISO-ACC Class -5	ISO-ACC Class -6	ISO-ACC Class -6
Planetary Sample Handling Rm. 2 (PSHR2)	ISO-ACC Class -4	ISO-ACC Class -6	ISO-ACC Class -6
Electron Microscope Room (EMR)	ISO-ACC Class -4	ISO-ACC Class -6	ISO-ACC Class -6
Manufacturing and Cleaning Room (MCR)	ISO-ACC Class -4	ISO-ACC Class -6	ISO-ACC Class -5

*1: The lowest class of each element is shown in the table.

Table 3 Cleanliness classification of the surface cleanliness by chemical contamination for each location (ISO-14644-10)

Location		Surface cleanliness by chemical concentration (SCC)	
		Total organic matter	**Metallic elements*1**
Planetary Sample Handling Rm. 1 (PSHR)	Inside of the room	ISO-SCC Class -8	ISO-SCC Class -10
	CC2	ISO-SCC Class -9	ISO-SCC Class -10
Planetary Sample Handling Rm. 2 (PSHR2)	Inside of the room	ISO-SCC Class -8	ISO-SCC Class -10
	CC3-3	ISO-SCC Class -9	ISO-SCC Class -10
	CC4-1	ISO-SCC Class -9	ISO-SCC Class -10
	CC4-2	ISO-SCC Class -9	ISO-SCC Class -10
	Desiccator	ISO-SCC Class -8	ISO-SCC Class -10
Electron Microscope Room (EMR)	Inside of the room	ISO-SCC Class -8	ISO-SCC Class -10
	GB1	ISO-SCC Class -8	ISO-SCC Class -10
	GB6	ISO-SCC Class -8	ISO-SCC Class -11
	GB7	ISO-SCC Class -8	ISO-SCC Class -12
Manufacturing and Cleaning Room (MCR)	Inside of the room	ISO-SCC Class -8	ISO-SCC Class -10
	SUS clean booth	ISO-SCC Class -8	ISO-SCC Class -10
	Resin clean booth	ISO-SCC Class -8	ISO-SCC Class -10

*1: The lowest class of each element is shown in the table.

3.2 In-house organic analysis

In qualitative analysis using GC×GC-TOFMS, the qualitative ability has been significantly improved, and more than 1000 peaks could be detected in a single analysis, and approximately 400 types of components can be identified (similarity: >900). We implemented the in-house organic analysis from March 2022 in EMR to assess the effects of construction of a new clean room on the cleanliness of EMR where these two rooms are adjacent to each other. The constructions lasted for five months between June and October 2022. We also have performed this analysis in the other clean rooms periodically to monitor their cleanliness levels: PSHR2 starting from July 2022 and PSHR, MCR, SPR2 and SPR3 from August 2022.

Though EMR is the only room where we conducted the organic analyses three times over several months, the intensity profile of the total organics in EMR shows the nearly double-increase post construction of the new clean room (Fig. 7). We observed that increased organic compounds primarily consisted of toluene (Benzene, methyl-), xylene (Benzene, 1,3-dimethyl-) and cyclohexane. Figure 8 shows the organic compounds of particularly high intensities. They are commonly used in paints, adhesives, sealing materials, rubber solvent and so on, which implies that the construction work of a new clean room (PSHR3) may have affected on the cleanliness in adjoining clean rooms. Floor painting was carried out on August 12th in 2022, and organics were sampled before and after the painting to analyze the painting effects on the cleanliness level of PSHR. Total organic compounds increased immediately after the painting. Organics in the newly constructed SPR2 and SPR3 were analyzed and their intensities were the same level as PSHR2 (Fig. 7).

※1 Construction of PSHR3 started from 2022/7/1

※2 Painting was done as a floor repair in the PSHR on 2022/8/12

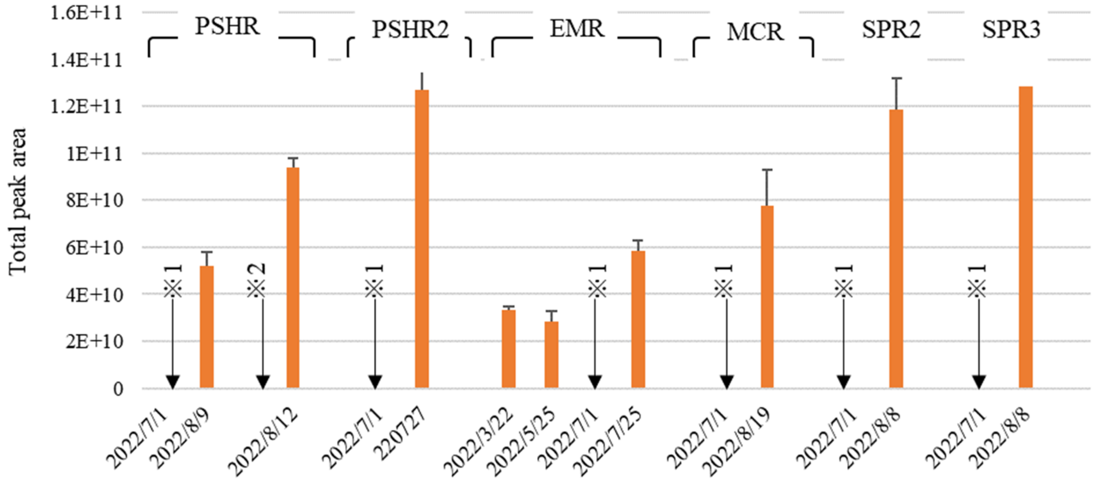

Fig. 7 The intensity profile of the total organic compounds in clean rooms

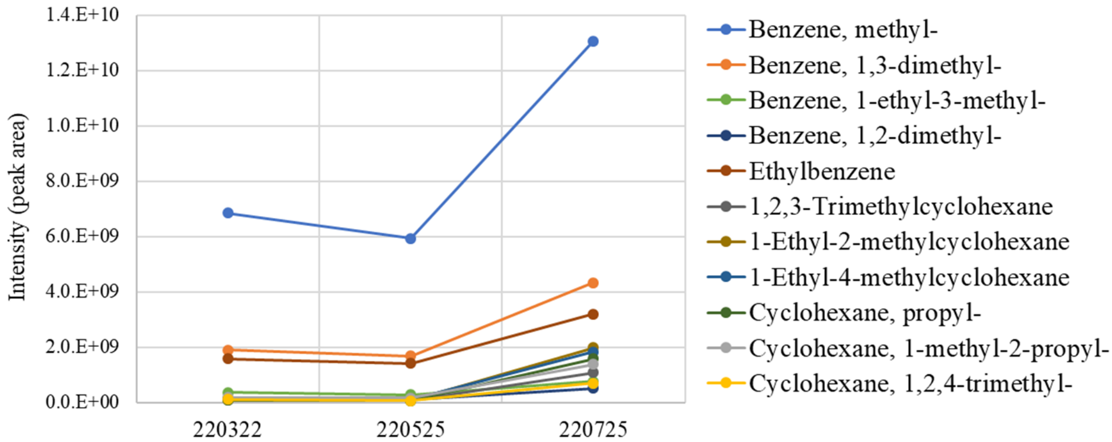

Fig. 8 Selected organic compounds showing the extreme increase of each compound by PSHR3 construction work

3.3 Microbial contamination analysis

The results of colony count on culture media in different cultivating conditions are shown in Table 4. No microbial contaminations were detected in all the media collected from both PSHR2, and CC4-1.

Table 4 Results of cultivation-based analyses

Media (quantity of media)	**Colonies number**				
	P.C.	**N.C.**	**CC4-1 floor**	**CC4-1 glove**	**PSHR2 floor**
TSA plate (4)	2, 1, 0, 0	0, 0, 0, 0	0, 0, 0, 0	0, 0, 0, 0	0, 0, 0, 0
Blood agar plate (2)	2, 0	0, 0	0, 0	0, 0	0, 0
R2A plate (2)	3, 1	1, 0	0, 0	0, 0	0, 0
Saboraud-dextrose plate (1)	1	0	0	0	0
Saboraud-dextrose with chloramphenicol plate (2)	3, 1	0, 0	0, 0	0, 0	0, 0
Poteto-dextrose plate (1)	2	0	0	0	0
Thioglycolate plate (2)	1, 0	0	0	0	0

3.4 Future activities

In this study, we present the in-house organic analysis data as intensity of chromatograph because a protocol of quantitative analysis has not been established yet. It is important to compare periodical in-house and annual outsourced contamination analysis data for a quantitative study. We are currently considering a quantitative method using standard substances. The outgas collecting instrument (FD-4000DF, Japan Analytical Industry Co Ltd.) was procured and we will set up this instrument to analyze organic samples collected inside the clean chambers by the wafer exposure method. The current in-house analysis is limited in measuring only organics. We are planning to expand the sample category from organics to metallic elements, using ICP-MS combined with Gas Exchange Device (GED) that allows to analyze nano particles (metallic elements) in the air in the clean rooms, and are proceeding with the upgrading utilities in the clean room (MCR). GED is an introduction system that enables direct collection of gas samples including nano particles into ICP-MS for the direct analyses of their metallic particles. This will allow us to obtain more comparable data with the annual contamination analysis data for a comparative study.

The microbial analysis is also one of the important methods to evaluate contamination. Colony counting method can be used to detect only living and culturable microbes. Since 99% of microorganisms are known not to grow on the cultivation plates, we further plan to conduct DNA based analysis using with the same samples to detect unculturable microorganisms.

4 SUMMARY

The ESCuC has been conducting organic and inorganic contamination analyses for the clean rooms, clean chambers and part of the glove boxes annually by outsourcing since 2013. We compared the analysis data obtained in 2021 and partly in 2022 with those before the Ryugu sample returned to the earth in December 2020. The results revealed that the cleanliness level of the clean rooms and clean chambers are maintained at similar level after the Ryugu sample return. Only sodium and potassium concentrations mainly derived from human bodies are significantly higher than before, and this is considered to be because of the increased curatorial activities such as processing and analyzing of Ryugu samples after the sample return. The contamination analysis was also conducted for the glove boxes newly installed in 2022 (GB6 and GB7). The total organic contents in GB6 and GB7 are less than the other glove box (GB1) and metal elements in the two glove boxes are mostly below the blank level as GB1.

In addition to the annual contamination analysis, we installed GC×GC-TOFMS to analyze organic contaminants more frequently with flexible timing. Air samples were collected in the clean rooms and analyzed mainly focused on monitoring the environmental change after starting the construction of the new clean room (PSHR3), and also influence of the floor painting in PSHR1. The total organic contents in EMR next to the constructing room significantly increased after starting the construction, and increased organic materials are commonly used in paints, adhesives, sealing materials, rubber solvent and so on, which strongly implies the influence of new clean room construction.

Microbial contamination analysis, which is also one of the essential tests for monitoring contaminants, was conducted using the samples collected in a clean room (PSHR2) and a clean chamber (CC4-1). Results of colony counting indicated no microbiological contaminations in the sampled areas.

We indicated here the efficiencies and advantages of in-house contamination monitoring. For the next step, we plan to install GED-ICP-MS in our curation facility to analyze contamination of not only organics but also inorganic materials. Establishing a of protocol for microbial contamination monitoring is also required in the near future.

ACKNOWLEDGEMENTS

The authors thank the team from The Renesas Semiconductor Manufacturing Co., Ltd. conducted the analyses in this study.

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JAXA Special Publication JAXA-SP-22-006E

Contamination analyses of clean rooms and clean chambers at the Extraterrestrial Sample Curation Center of JAXA in 2021 and 2022

Edited and Published by: Japan Aerospace Exploration Agency
7-44-1 Jindaiji-higashimachi, Chofu-shi, Tokyo 182-8522 Japan
URL: https://www.jaxa.jp/
Date of Issue: February 3, 2023
Produced by: Matsueda Printing Inc.

Japan Aerospace Exploration Agency