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The GAs Extraction and Analyses system (GAEA) for immediate extraction and measurements of volatiles in the Hayabusa2 sample container

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Abstract

Hayabusa2 returned surface samples from the C-type near-Earth asteroid (162173) Ryugu to Woomera, South Australia, in December 2020. The samples returned from Ryugu are expected to contain not only volatile components reflecting its primitive nature, but also solar wind components due to exposure to space. Such volatiles may partly be released inside the sealed sample container enclosing Ryugu samples due to particle destruction or container heating in a contingency case. In order to collect and analyze volatiles released in the container prior to the container-opening, we set up a gas extraction and analyses system (GAEA: GAs Extraction and Analyses system). The system requires ultra-high vacuum conditions, small vacuum line volume to minimize dead volume and simple configuration as well as having an interface to connect the container. The system includes gas bottles for passive collection of volatiles at room temperature and bottles for active collection at liquid nitrogen temperature. A quadrupole mass spectrometer is installed to analyze gases in the vacuum line, and a non-evaporative getter pump is also used when noble gases are analyzed. The rehearsal operation of the GAEA was made at ISAS/JAXA as well as transportation tests in Japan. In November 2020, it was transported safely to the Quick Look Facility (QLF) in Woomera. It was set up at the QLF and worked as planned for collection and analysis of gas components from the returned sample container. Here we report the concept, design and calibration results for the GAEA and an outline of analytical protocols applied in Woomera.

Keywords: Hayabusa2, Sample return, C-type asteroid, Volatiles, Gas analysis

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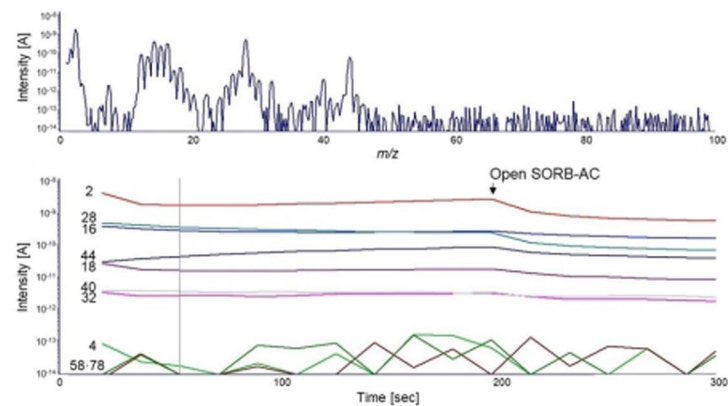
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Graphical Abstract



Calibration air #10 (c)



Introduction

Hayabusa2 is the second asteroidal sample return mission operated by the Japan Aerospace Exploration Agency (JAXA). It launched on December 3, 2014 and arrived at the target asteroid (162173) Ryugu on June 27, 2018. Ryugu is a C-type near-Earth asteroid covered with regolith that also contains pebbles and sands (Tachibana et al. 2022 and references therein). C-type asteroids have been considered to record the early evolution of the Solar System in their primitive constituents including volatiles (Tachibana et al. 2014; Watanabe et al. 2017). Science objectives of the Hayabusa2 mission are to investigate (1) the evolution from a planetesimal to a near-Earth asteroid (i.e., thermal processes in a planetesimal in the early solar system, heating and space weathering on the surface of near-Earth asteroid at its current orbit); (2) potential destruction and accumulation of a rubble-pile body that formed from a larger aqueously altered parent planetesimal (i.e., planetesimal formation and impact processes throughout the solar system history); (3) diversification of organic materials through interactions with minerals and water in a planetesimal (i.e., origin and evolution of volatile components in the early solar system and final state of organic matter and water prior to their delivery to the rocky planets) and (4) chemical heterogeneity in the early solar system (i.e., mixing of high-temperature and low-temperature components during dynamical evolution of the proto-solar disk) (e.g., Tachibana et al. 2014; Watanabe et al. 2017). In order to clarify these objectives, the volatile components are very important targets to be analyzed on ground. It has been known that primitive meteorites contain water and a variety of organic matters synthesized from volatile elements such as H, C, N, and O. Such water and organics could have been related

to a possible source of Earth's ocean and life. In addition, implanted solar wind and galactic cosmic-ray produced nuclides are likely exist, similar to the returned samples from the asteroid Itokawa (Nagao et al. 2011; Busemann et al. 2015) and lunar samples (Wieler 2016 and references therein), from which surface histories such as gardening efficiency and time scale of movement of surface material could be constrained. Small fractions of solar wind noble gases were released at low temperatures like 100 °C for lunar samples and gas-rich meteorites (e.g., Black 1972a, 1972b; Pepin et al. 1999), therefore, not only components kept in solid samples, but also those released into gas phases in the sample container should be recovered and analyzed.

In order to reduce terrestrial contamination, we planned that the volatile gases in the sample container are collected in gas bottles and measured with a quadrupole mass spectrometer (QMS) soon after the recovery of the Sample Return Capsule (SRC) in Australia. The Hayabusa2 sample container has a metal-to-metal sealing system and a gas-sampling interface to extract volatiles without exposure to the terrestrial atmosphere, which were developed based on the lessons learned from the Hayabusa mission (Okazaki et al. 2017; Sawada et al. 2017). Another motivation of gas extraction from the sample container before opening is to keep the interior pressure as low as possible to avoid the loss of sample due to gas stream induced pressure differences between the container and the vacuum clean chamber dedicated to Hayabusa2 samples at ISAS/JAXA. This motivation also came from the lessons learned from the Hayabusa mission, where the poorly constrained container pressure prevented prompt container-opening in a clean chamber (Okazaki et al. 2011).

Hayabusa2 carried out remote sensing observations on Ryugu with findings of OH-related absorption and also touchdown operations at two landing sites for sample collection (e.g., Sugita et al. 2019; Arakawa et al. 2020; Morota et al. 2020; Tsuda et al. 2020; Kameda et al. 2021; Kitazato et al. 2021; Okada et al. 2021; Tatsumi et al. 2021; Tachibana et al. 2022). Then, Hayabusa2 left Ryugu on November 13, 2019 (Saiki et al. 2021). After its journey towards Earth, the spacecraft successfully delivered the SRC, which landed at Woomera desert in Australia on December 6, 2020 (Yamada et al. 2021). The SRC was found ~2 h after its landing and immediately carried to the Quick Look Facility (QLF) in the Woomera Prohibited Area (WPA), South Australia (Tachibana et al. 2021). The sample container, in which the collected samples were stored, was taken from the SRC and set on a base-plate of the ICF356 flange (Fig. 1). Then, it was connected to the GAs Extraction and Analyses system (GAEA) via an ICF34 flange of the metal valve in order to extract, collect, and measure volatile components in the sample container before the container-opening at ISAS/JAXA in Sagamihara, Japan. After these operations in the WPA, the sample container was kept as the static condition during transportation to ISAS/JAXA. In this paper, (i) the

concept and configuration of the GAEA and (ii) an outline of the analytical procedure at the QLF in Woomera are described.

Gas Extraction and Analyses system (GAEA)

System concept and requirements

The entire system was designed based on the concepts of (a) keeping an ultra-high vacuum condition using oil-free pumps; (b) small line volumes to minimize dead volume; (c) introducing a programmable valve control system to avoid human errors and protect against accidental power failure, and (d) simple, firm, and easy-handling configuration to be transported between Japan and Australia. The system was required to fit within 1.5 m × 1.5 m × 1.5 m in size and to be operated with 10 kVA. As basic performance, dynamic (non-static) pressures of analytical lines were requested to be $<1 \times 10^{-5}$ and $<1 \times 10^{-8}$ Pa at the gas extraction line and near turbo molecular pumps, respectively, within 150 h after re-starting pumps. These requirements are important to enable certainly and promptly the volatile element analysis for Hayabusa2 container gases in Australia within limited time, space, and power supply. In order to configure the system that

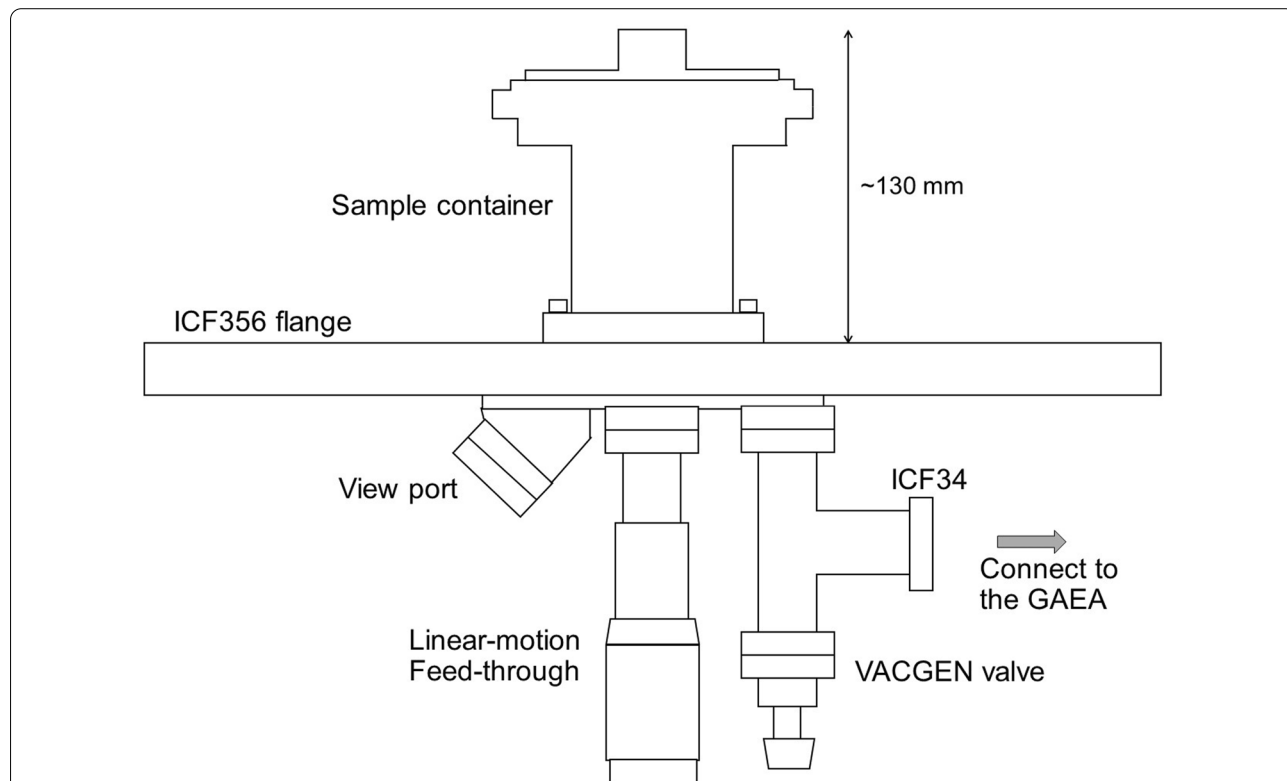


Fig. 1 Schematic drawing of the Hayabusa2 sample container mounted on the ICF356 flange. The flange is equipped with a linear motion feed-through, a view port, and a metal valve (VACGEN ZCR20). It is connected to the GAEA via the ICF34 port of the valve

satisfies the requirements, we performed some preliminary experiments and then designed the system.

Development and preliminary experiments

We developed an original gas extraction method, that is gases are extracted from the sample container without exposure to the terrestrial atmosphere by making a hole at the bottom of the container (HAWKS; HAYabusa2 gas collection With the Keyhole System). The principle of the HAWKS was reported in Okazaki et al. (2017). Schematic drawings of the gas-sampling interface of the sample container and the final design of the needle are shown in Fig. 2. Our experiments conducted before the launch of Hayabusa2 indicated that a hole can be made without any leakage in the sealing between the container and the base flange and without any mechanical troubles such as stacking. For more secure operation to be performed in Woomera, we reexamined suitable shapes of the tungsten

carbide needle and feed stroke of the needle. Because the clearance between the bottom plate of the container and a filter set above the plate is ~0.85 mm (Fig. 2c), the moving distance by the linear feed-through must be controlled to an accuracy far better than ± 1 mm not to break the filter. Ten designs of needles with different sizes were prepared and tested: two types of a corn head angle (30 and 45°), three types of corn head sharpness (sharp, 0.3 and 0.5 mm in diameter), four sizes of cylindrical shape heads at the top of the corn heads (0.3, 0.5, 0.6, and 0.7 mm in diameter) and some combinations of them. Test results showed somewhat different hole morphologies. Among them, a needle piercing a hole most flat and smooth was selected for the actual operation (Fig. 2d). Its body diameter is 3 mm, and the corn head angle is 30°. The tip of the needle has a delta corn shape with a pin-like protuberance (0.6 mm in height and 0.6 mm in diameter). The stroke of moving the needle, by measuring the

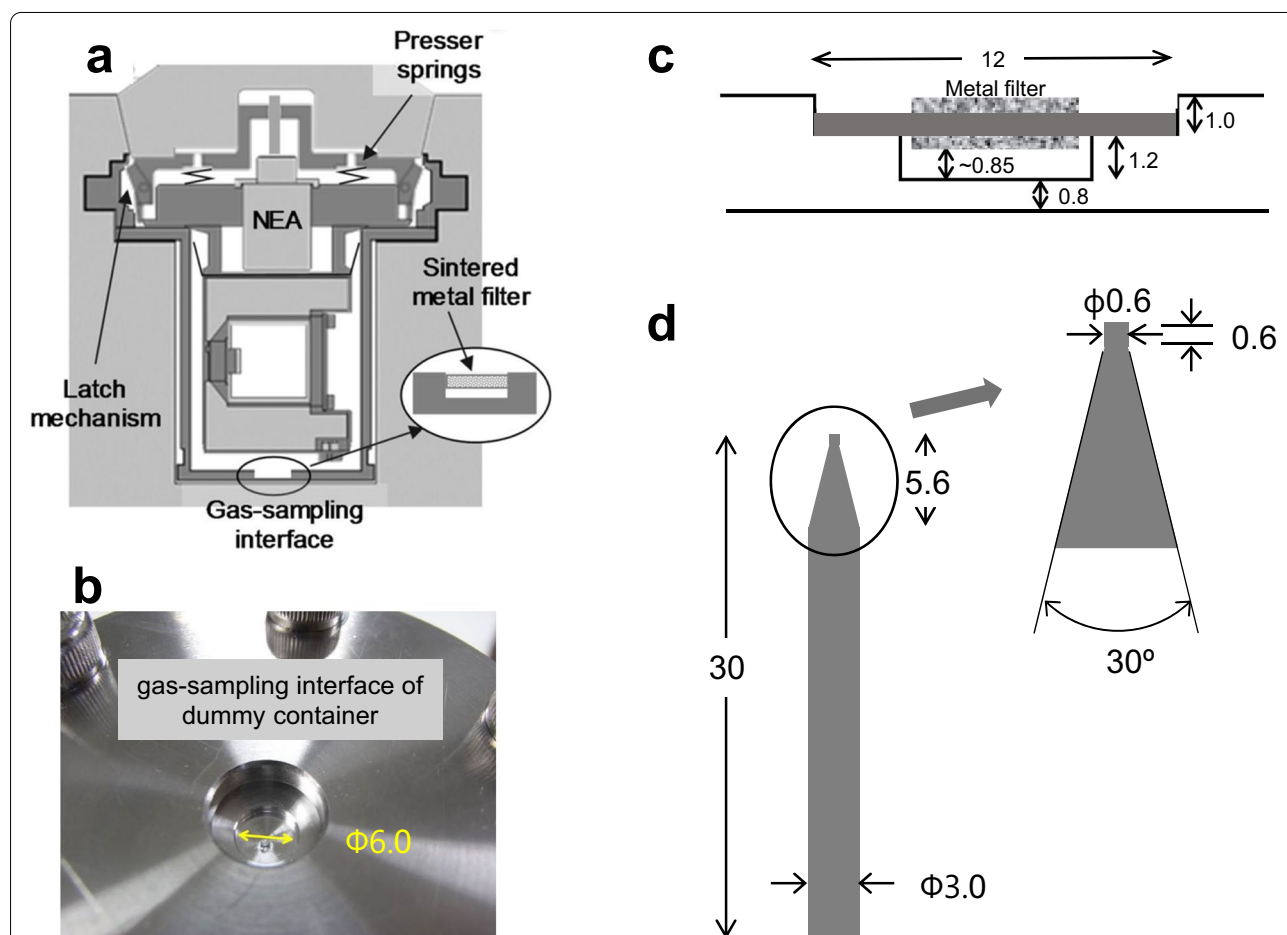


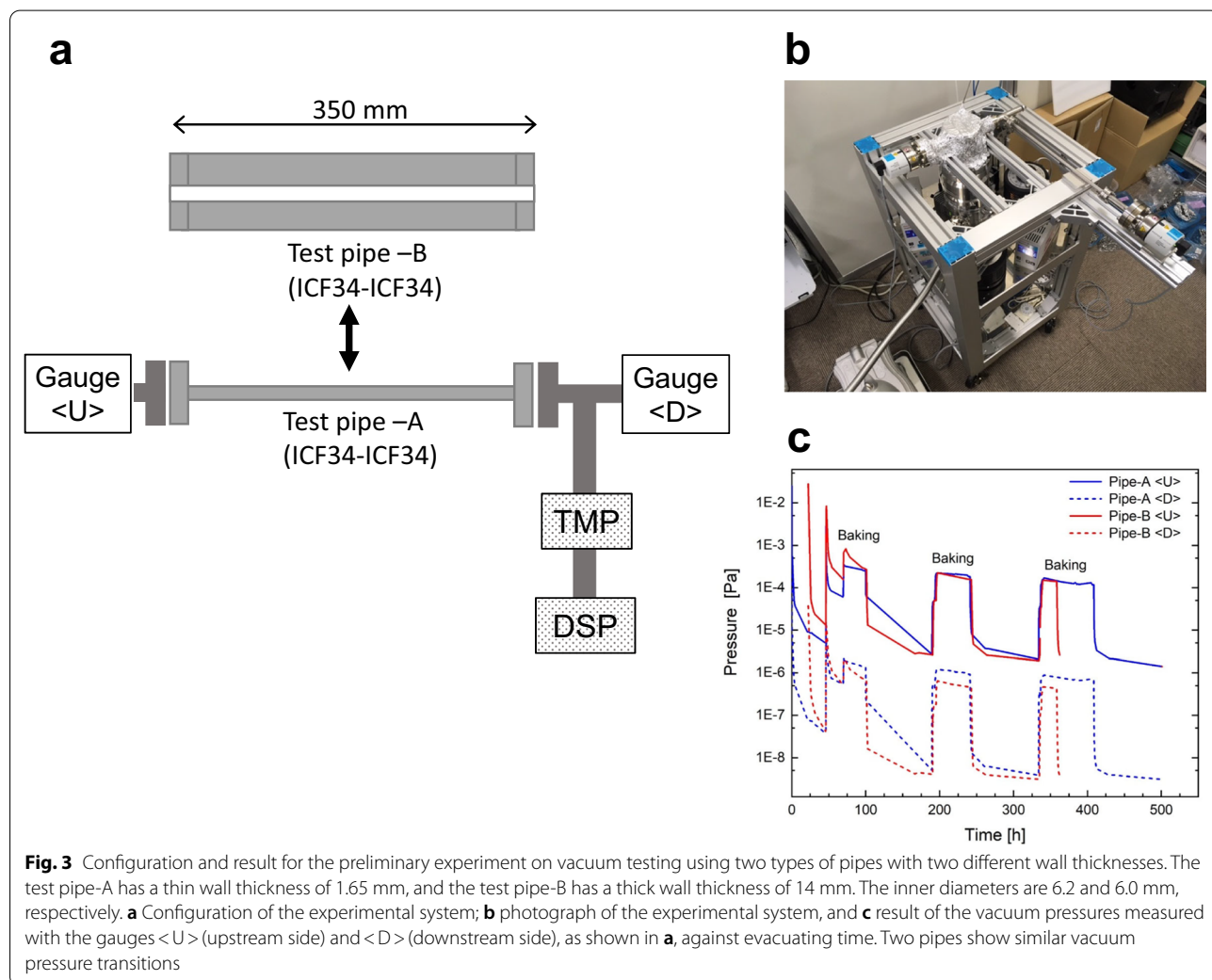
Fig. 2 Gas sampling interface and needle for the Hayabusa2 gas collection with the Keyhole System (HAWKS). **a** Cross section of the Hayabusa2 sample container (after Okazaki et al. 2017); **b** photograph for the gas-sampling interface of a dummy container; **c** dimension for the gas-sampling interface at the bottom of the sample container, where the metal filter adopted here is Swagelok SS-4-VCR-2-.5 M (made of SS (stainless steel) 316 and the defined pore size is 0.5–2 μm), and **d** schematic drawing of the needle that we used for the actual operation at Woomera. The numbers are the sizes given in millimeters

micro-scale of the feed-through, was determined to be 1.2 mm through preliminary tests using simulated sample containers. The final GAEA design allowed us to extract gases successfully using HAWKS in Woomera.

The Hayabusa2 sample container has approximately 200 cm³ in volume. To collect most of the gases stored in the sample container (e.g., ~90% of the gases) into a reasonable size of four bottles, the line volume of gas-expanding-areas must be ≤ 100 cm³. For this, we considered to use pipes with small diameter. However, because pipes with small diameter have smaller conductance, pumping efficiency was a matter of concern. Therefore, we examined pumping efficiency and ultimate vacuum pressure test using pipes with two different inner diameters (ID) of ~11 and ~6 mm. The two test pipes that were a few ten cm long with three corners, each corner reducing pumping speed, showed an order of magnitude different vacuum pressures at the gauge sited in the vacuum lines. We also tested if there is a pressure difference by

the effect of hydrogen permeation using small ID pipes having different pipe-wall thicknesses of ~1.7 mm and 14 mm. The results of the vacuum pressures at the end of each pipe are given in Fig. 3, showing no notable difference. After bake-out at 200 °C several times, the pressures reached at the end of these pipes are ≤ 5 × 10⁻⁶ Pa (Fig. 3c). The hydrogen permeation into the line throughout the pipe wall was not a problem even for the small-bore pipe with the thin wall thickness. Considering these observations, we designed the GAEA predominantly using small ID pipes (~6.5 mm) with thin wall thickness (~1.2 mm) for main pipes with straight line structure.

For pressure measurements, cold cathode gauges (CCG) are used near the turbo molecular pumps because of their quick response and easy usage for monitoring wide range pressures. A miniature ion gauge (MG) and a Pirani gauge (PG) are chosen at a gas-expanding-area to measure pressures of gases released from the sample container. The CCGs have relatively low accuracy (accuracy



decreases with operation time) due to cathode sputtering and plasma-chemical reactions that causes contamination within the gauge (Wilfert 2003) and no function to avoid such contamination. We also confirmed that the gas pressures measured by the CCG decreased more rapidly than those by the MG for calibration air. Therefore, we measure the pressure of sample gas with the PG, and then, if the pressure is lower than the measurable range of PG (≤ 0.4 Pa), we turn on the filament of MG. In order to avoid the effect like decomposition and implantation of ionized gases, we kept the duration of filament-ON as short as possible during exposure to the gas, that is typically for 1 to a few seconds.

A quadrupole mass spectrometer (QMS) is used for identification of volatile constituents. QMS has advantages of easy operation and quick measurements. We also preliminarily examined background mass spectra for three commercial QMS, among which one (Tokyo Electronics, Watmass MPH200M) that exhibited the lowest background of residual mass spectra was

selected. In addition, we introduced another QMS for leak testing after connecting the sample container.

As mentioned, one of the goals was to analyze solar wind noble gases, particularly He and Ne. In order to remove active gases, a SORB-AC getter (SAES non-evaporative getter pump; hereafter SORB-AC) is used. It can be used before or during QMS measurements by opening a valve. It is also helpful to evaluate contributions of active gases, such as hydrocarbon, nitrogen, and oxide molecules, by comparing mass spectra with and without SORB-AC. Essentially, only noble gases are not trapped by SORB-AC, which means peak intensities of noble gases are similar between with and without SORB-AC.

Instrument descriptions

A schematic figure and an external view of the GAEA are shown in Figs. 4 and 5, respectively. The system is composed of:

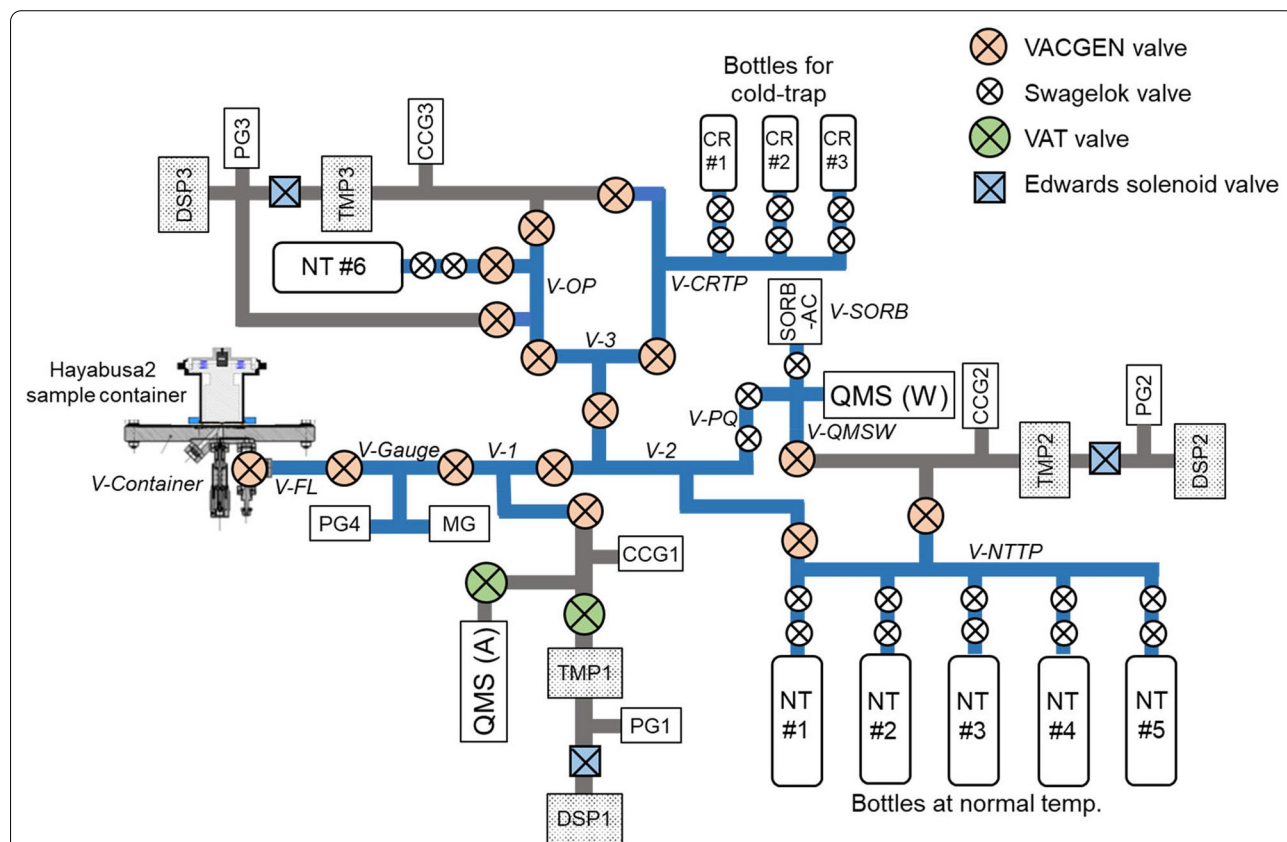


Fig. 4 Schematic drawing of the GAs Extraction and Analyses system (GAEA). Working volumes (areas used for gas expansion) are shown in blue and pumping lines are shown in gray. Each area between valves is named as labeled in italic. Abbreviation: QMS (W)—quadrupole mass spectrometer (Watmass), QMS (A)—quadrupole mass spectrometer (Anelva), SORB-AC—SORB-AC getter, CCG—cold cathode gauge, MG—miniature ion gauge, PG—Pirani gauge, TMP—turbo molecular pump, DSP—dry scroll pump

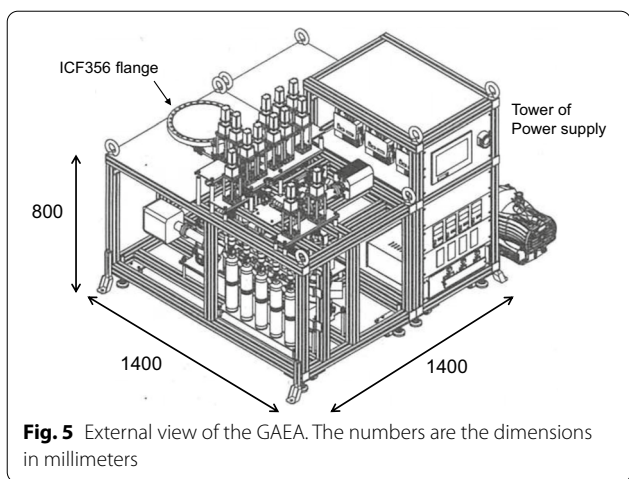


Fig. 5 External view of the GAEA. The numbers are the dimensions in millimeters

- metal lines (mostly made of SS 316) and all metal valves (VACGEN ZCR20 and Swagelok SS-4H) for ultra-high-vacuum,
- a connecting port to attach the Hayabusa2 sample container that is mounted on an ICF356 flange with an ICF34 port (Fig. 1),
- pressure gauges to monitor extracted gases and vacuum conditions of the system (Ulvac GP1000G, Anelva MG-2, and Pfeiffer IKR270),
- a SORB-AC getter (SAES NP-10),
- six bottles for storing the container gases at room temperature (bottles at normal temperature, a design drawing is given in Additional file 1: Fig. S1a) and three bottles for trapping the gases at the liquid nitrogen temperature (bottles for cold-trap, Additional file 1: Fig. S1b), among them each one bottle is used to collect background gases of the GAEA at room temperature and at the liquid nitrogen temperature,
- two quadrupole mass spectrometers (QMS): (i) QMS (W); main QMS for container gas analyses (Tokyo Electronics, Watmass MPH 200 M, for which Inficon

Transpector MPH200M is used as the base model and the operation software is Inficon FebGuard Explorer), and (ii) QMS (A) used for leak testing (Anelva M-201QA-TDM),

- three vacuum pump sets, each of which consists of a turbo molecular pump (Shimazu TMP-B300), a dry scroll pump (Edwards nXDS10i), a solenoid operation isolation valve (Edwards PV25EK), and a pirani gauge (Ulvac PG1000G),
- a power supply controller,
- motor drive devices for metal valve (ZCR20) operation that developed by R-DEC Co., Ltd. (ZCR20 operatable using a motor drive or on manual, and SS-4H on manual),
- heaters and controllers to bake-out the vacuum lines.

The main power voltage for the system is 100 V except for baking heaters and motor drives to operate the ZCR20 valves. The power supply distribution is schematically drawn in Fig. 6. Transformers to transform the voltage from 240 to 100 V are prepared for operation in Australia. Three UPSs (uninterruptible power supplies; GS Uasa THA5000-10 and Omuron BU200RW) are also provided as backup power sources for any accidental power cut. For system safety, all the ZCR20 valves are automatically closed with alerts for power cut (a UPS starts) or unusual decrease of a vacuum pressure.

The line volumes used for gas expansion, colored in blue on Fig. 4, were determined by a method based on Boyle’s law. Two vessels with known volumes (107 and 205 cm³) were used as references. The pressure difference was measured between [pipe of unknown volume] and [pipe of unknown volume + one of the reference vessels] by a manometer. The pressure of N₂ gas used for manometry was first adjusted to be ~150 Pa. The volumes of each line area are summarized in Table 1. The gas-storage

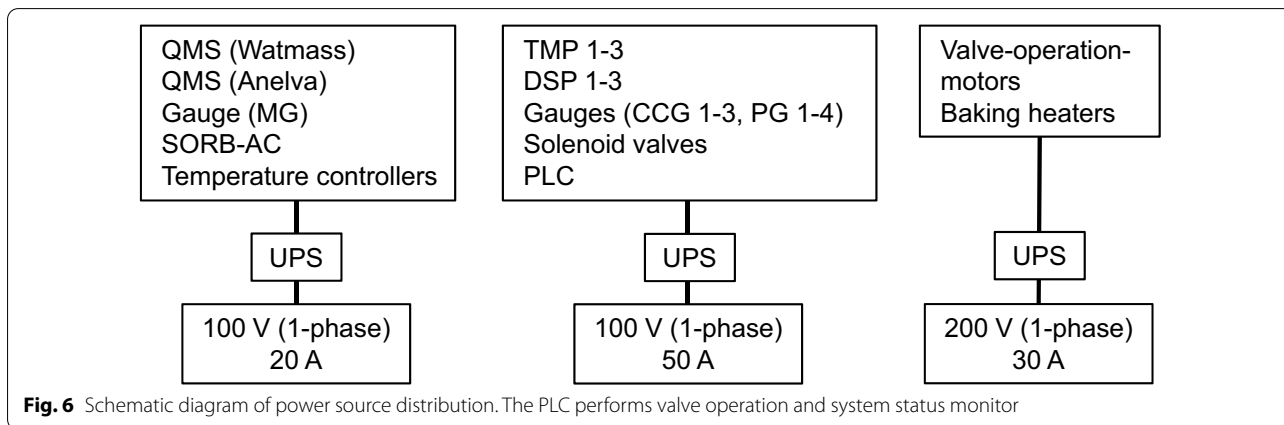


Fig. 6 Schematic diagram of power source distribution. The PLC performs valve operation and system status monitor

Table 1 Volumes of each gas-expanding area of the GAEA and gas bottles collecting Hayabusa2 container gases

Area name	Volume (cm ³)	Gas bottle	Volume (cm ³)
V-Container	200	Bottles at normal temperature	
V-FL	15	NT #1–#6	750
V-Gauge	64	P-NT #1–#6	40
V-1	31.9	Bottles for cold-trap	
V-2	36.4	CR #1–#3	50
V-3	36.5	P-CR #1–#3	16
V-PQ	15.5	P-: pipet attached to the bottle	
V-SORB	25.6		
V-QMS (cross)	33.3		
QMS (W)	296		
V-QMSW [#]	329		
V-NTTP	45.4		
V-OP	53.7		
V-CRTP	46.4		

See Fig. 4 for area definition

[#] V-QMSW = V-QMS (cross) + QMS (W)

Volumes were determined with the valve position of nearly close. Shown volumes may slightly change (by ~ 1 cm³) depending on the valve position (close/open)

bottles at normal temperature (NT #1–#6) and those for liquid nitrogen temperature (CR #1–#3) have the volumes of 750 and 50 cm³, respectively (Additional file 1:

Fig. S1 for their design drawings). The Hayabusa2 sample container and a vacuum flexible bellows that connects between the container and the GAEA have approximate volumes of 200 and 15 cm³, respectively. Gas reduction factors are calculated using these volumes (Table 2), based on which we evaluate the original gas amount inside the sample container. Among the extracted gases from the container, 80% is stored in four bottles of NT #1–#4 (20% in each bottle). Remaining gases are used for the measurements with QMS (W), the collection in two bottles of CR #1–#2 as cold-trap gases at the liquid nitrogen temperature and are re-analyzed (stored in NT #6 and re-measured with QMS (W)).

Pre-operation and calibration at ISAS

The GAEA was installed at the Sagamihara campus of ISAS/JAXA in 2019. Photographs of the GAEA set at the ISAS/JAXA (Sagamihara, Japan) and the QLF (Woomera, Australia) are shown in Fig. 7 and Additional file 1: Fig. S2. After confirmation of the initial condition, the system was moved to Kyushu University for improvements and a transportation test on November 28, 2019. The system was transported safely back to ISAS/JAXA on June 22, 2020 and was set up again for the operation test and training. From June to September 2020, bake-out of the system, calibration and some adjustments for stable operation were carried out. The

Table 2 Reduction factors of gases for measuring and collecting Hayabusa2 container gases using the GAEA

Description	Reduction condition	Fraction
Gases in the container after expanding to V-Gauge	(V-Container/V-Container + V-FL + V-Gauge)	0.717
Gases kept in V-PQ for the QMS measurement ^a	(V-PQ/V-Container + V-FL + V-Gauge + V-1 + V-2 + V-PQ)	0.0428
Gases collected in a NT bottle (each of NT #1–#4) ^b	(V-Container + V-FL + V-Gauge + V-1 + V-2/V-Container + V-FL + V-Gauge + V-1 + V-2 + V-PQ) × (NTbottle/V-Container + V-FL + V-Gauge + V-1 + V-2 + V-NTTP + 4* NTbottle + 4*P-NTbottle)	0.202
Gases collected in CR #1 ^c	(V-Container + V-FL + V-Gauge + V-1 + V-2/V-Container + V-FL + V-Gauge + V-1 + V-2 + V-PQ) × (V-Gauge + V-1 + V-2 + V-NTTP/V-Container + V-FL + V-Gauge + V-1 + V-2 + V-NTTP + 4* NTbottle + 4*P-NTbottle) × (CRbottle/V-Gauge + V-1 + V-2 + V-3 + V-NTTP + V-CRTP + CRbottle + P-CRbottle)	0.00736
Gases collected in CR #2 ^c	(V-Container + V-FL + V-Gauge + V-1 + V-2/V-Container + V-FL + V-Gauge + V-1 + V-2 + V-PQ) × (V-Gauge + V-1 + V-2 + V-NTTP/V-Container + V-FL + V-Gauge + V-1 + V-2 + V-NTTP + 4* NTbottle + 4*P-NTbottle) × (V-Gauge + V-1 + V-2 + V-3 + V-NTTP + V-CRTP + V-CRTP/V-Gauge + V-1 + V-2 + V-3 + V-NTTP + V-CRTP + CRbottle + P-CRbottle) × (V-CRbottle/V-Gauge + V-1 + V-2 + V-3 + V-NTTP + V-CRTP + CRbottle + P-CRbottle)	0.00588
Gases collected in NT #6	V-Container + V-FL + V-Gauge + V-1 + V-2/V-Container + V-FL + V-Gauge + V-1 + V-2 + V-PQ) × (V-Container + V-FL/V-Container + V-FL + V-Gauge + V-1 + V-2 + V-NTTP + 4*NTbottle + 4*P-NTbottle) × (V-Container + V-FL + V-Gauge + V-1 + V-2/V-Container + V-FL + V-Gauge + V-1 + V-2 + V-PQ) × (NTbottle/V-Container + V-FL + V-Gauge + V-1 + V-2 + V-3 + V-OP + NTbottle + P-NTbottle)	0.0339

See Fig. 4 and Table 1 for the names and volumes

Fractions are calculated using the volumes shown in Table 1

^a Gases kept in V-PQ are reduced before introducing into the QMS if the gas pressure is too high

^b Fraction of gases collected in each bottle of NT #1–#4

^c Fraction of gases collected in CR #1 or CR #2 for the case assuming at normal temperature



Fig. 7 Photographs of the GAEA at the ISAS/JAXA (Sagamihara) and the QLF (Woomera)

regular vacuum pressures reached down to 1×10^{-8} , 5×10^{-9} and 7×10^{-9} Pa for CCG1, CCG2 and CCG3, respectively (see Fig. 4 for the system layout).

The sensitivity of the QMS (W) was determined for ^{40}Ar by introducing air into the system. Air is a mixture of 78 vol% of N_2 , 21 vol% of O_2 and 0.93 vol% of Ar, water vapor and some other gases as minor components. Argon has three stable isotopes ^{36}Ar , ^{38}Ar and ^{40}Ar , whose molar fractions in air are 0.003336, 0.000629 and 0.996035, respectively (Meija et al. 2016). The amount of air introduced in the system was required to be minimal to keep the background level as low as possible. For input of the gases into the system, a small tank containing the quantitative amount of air was attached at the ICF34 flange next to the line named [V-Gauge], to which the Hayabusa2 sample container mounted on the ICF356-baseplate is connected via

[V-FL] (the flange port and [V-FL] had never been used before the actual operation in Woomera). The QMS (W) has two detector modes, the Faraday-cup mode (lower sensitivity) and the SEM mode (higher sensitivity). The SEM mode was used for measurements in nominal operation. The operating condition of the QMS (W) was 2 mA for the emission current, 70 eV for the electron energy and 900 V for the secondary electron multiplier voltage of the detector, respectively. The calibration data are summarized in Table 3. The amounts of the atmosphere introduced into the QMS (W) were 1.7×10^{-8} – 2.8×10^{-7} cm^3 STP (1.6×10^{-10} – 2.7×10^{-9} cm^3 STP for ^{40}Ar). The pressures in the QMS (W) during measurements were calculated to be 3.9×10^{-6} – 6.6×10^{-5} Pa based on the amounts of the gases and the expanding volumes. Representative mass spectra for the calibration (run numbers of 10(c), 11(a) and 12) and a blank run before the calibration 11(a) are shown in Fig. 8. In the calibration measurements, peak scans of m/z from 1 to 100 were conducted. The peak scan was started when gas was introduced into the QMS (W). The peak intensities were stabilized after the third scan, around 55 s after the gas introduction, and then decreased in general. The mass spectra were first obtained without SORB-AC, and after 2 min those were obtained with SORB-AC by opening the valve at SORB-AC (Fig. 4). The peak at $m/z = 40$ did not change largely between operation with and without SORB-AC (decrease by 0–25%), implying the peaks mostly to be ^{40}Ar . The amounts of ^{40}Ar in blank measurements were comparable to the detection limit ($\sim 2 \times 10^{-11}$ cm^3 STP) during the period of these calibration measurements. The mass spectra of the calibration and blank measurements show that there were no significant peaks higher than $m/z = 47$. For calculating the sensitivities, (i) the intensities of three data (around 50–90 s after the air introduction) were averaged, (ii) blank corrections were applied, and (iii) sensitivities were unified as the same volume condition of [V-PQ + V-QMS + QMS (W)] (Fig. 4) during the QMS measurements by volume corrections. The obtained sensitivities are listed in Table 3. The average of the sensitivities of ^{40}Ar was determined to be 520 ± 100 cm^3 STP/A by 12 measurements from July 8 to September 16, 2020. There is no dependence between the sensitivities and input amounts of air.

The intensities of $m/z = 28$ (I28) relative to $m/z = 40$ (I40) are also shown in Table 3. The peaks of $m/z = 28$ are possibly a mixture of N_2 and CO , because these are not resolvable for the QMS. The intensities shown in Table 3 were obtained by subtracting the intensities of blank measurements. Since the fraction of CO in air is much smaller than that of N_2 ($\text{CO}/\text{N}_2 < 10^{-5}$), the majority of gas

Table 3 Calibration condition, intensities of m/z 28 and 40, and sensitivity of ^{40}Ar

Calib. No	Date	Volume of meas. ^a	Pressure in QMS ^b (Pa)	Amount of ^{40}Ar in QMS (cm^3STP)	I40 (A)	I28/I40	Sensitivity of $^{40}\text{Ar}^c$ ($\text{cm}^3\text{STP/A}$)
8	7/8/20	PQ-	1.5E-05	4.37E-10	1.02E-12	120	428
9	7/14/20	PQ-	1.5E-05	4.33E-10	1.03E-12	136	421
10 (a)	7/21/20	PQ-	1.5E-05	4.29E-10	6.90E-13	95	621
10 (b)		V2-	3.1E-05	1.00E-09	1.99E-12	111	458
10 (c)		Vga-	6.6E-05	2.65E-09	4.05E-12	106	507
11 (a)	8/18/20	PQ-	1.5E-05	4.25E-10	6.23E-13	82	682
11 (b)		V2-	3.1E-05	9.96E-10	1.72E-12	89	524
11 (c)		V1-	2.5E-05	8.74E-10	1.30E-12	98	559
11 (d)		Vga-	4.4E-05	1.75E-09	2.47E-12	108	512
12	9/16/20	PQ-	1.5E-05	4.21E-10	8.77E-13	140	480
13 (a)		PQ-	6.0E-06	1.73E-10	2.50E-13	69	681
13 (b)		Vga-	3.9E-06	1.55E-10	3.19E-13	239	345
Average of all data:						116	518
						± 44	± 104
Average of data on 7/21 and 8/18:						98	540
						± 11	± 77

Terrestrial atmosphere was used as calibration gas. Volume fraction of ^{40}Ar is 0.0093

^a Volumes to which calibration gases were introduced for the QMS measurements

PQ-: [V-PQ + V-QMSW], V2-: [V-2 + V-PQ + V-QMSW], V1-: [V-1 + V-2 + V-PQ + V-QMSW], Vga-: [V-Gauge + V-1 + V-2 + V-PQ + V-QMSW]

^b Pressures in the QMS during analyses are calculated based on the gas amounts and the volumes shown in Table 1

^c Sensitivities are calculated using the average of three measured data around 50–90 s after inlet of gases into the QMS

All sensitivities are corrected as the analytical volume conditions to be [V-PQ + V-QMSW]

at $m/z=28$ is likely N_2 for the calibration gas. Although the observed ratios of I28/I40 were variable, calibration runs on July 21 and August 18, 2020, at which the blank contributions were relatively low, provided more consistent I28/I40 ratios with the average of 98 ± 11 . This ratio is close to the $\text{N}_2/^{40}\text{Ar}$ ratio of the terrestrial atmosphere ($\text{N}_2/^{40}\text{Ar}=84$; Ozima and Podosek 2002), suggesting that the relative sensitivity between N_2 and ^{40}Ar is close to 1 for the QMS (W). On the other hand, the signals for other molecules or elements were not significantly detected in the calibration measurements. Though O_2 is the second most abundant gas (the mole fraction is 0.209, that is ~ 22 times of Ar), the signal of O_2 ($m/z=32$) was almost similar to that for the blanks, which were 1–10 times that of Ar. The oxygen molecules were possibly dissociated at filaments of the QMS (W) or the ion pressure gauge, or alternatively, adsorbed on wall surfaces inside of the stainless-steel pipes via physicochemical reactions such as oxidation. Considering the intensities of $m/z=32$ being stable after gas introduction and no significant rise of the $m/z=32$ peaks, oxygen could have decreased before inlet into the QMS (W). Oxygen may react with hydrogen forming water that condenses on the internal surface, however, it is unclear what was the exact

mechanism at present. The empirical sensitivities relative to Ar were calculated to be 1.3, 1.5, 1.2, and 1.0 for N_2 , CO, O_2 , and CO_2 , respectively, according to previous studies on ionization probabilities, fragmentation factors and transmitting efficiencies of a quadrupole mass filter (cf., Summers 1969; Lieszkovsky et al. 1990; Jousten 2008; Malyshev and Middleman 2008; Yoshida and Arai 2018; Operating manual of Transpector MPH gas analysis system, PN 074-555-P1C, Inficon). The relative sensitivities reported for QMS in previous studies indicate more or less variation; those are within $1 \pm \sim 50\%$ for the species mentioned above in most cases (e.g., Mahaffy et al. 2011; Hirashita et al. 2013; Yoshida et al. 2014; Sun et al. 2020). We here consider the relative sensitivity factors for major gaseous species (except for O_2) to be 1 with the 50% uncertainty, which may include uncertainties arisen from long term stabilization of sensitivities, pressure dependence, cracking parameters of molecules during mass spectrometry. At least for nitrogen, the measured I28/I40 ratio of 98 provides the relative sensitivity factor to be 1.2, which agrees with the empirically calculated value shown above (1.3 for $\text{N}_2/^{40}\text{Ar}$). Nevertheless, we will basically indicate the notations as intensity ratios, like I28/I40, for measured gas compositions. The compositions

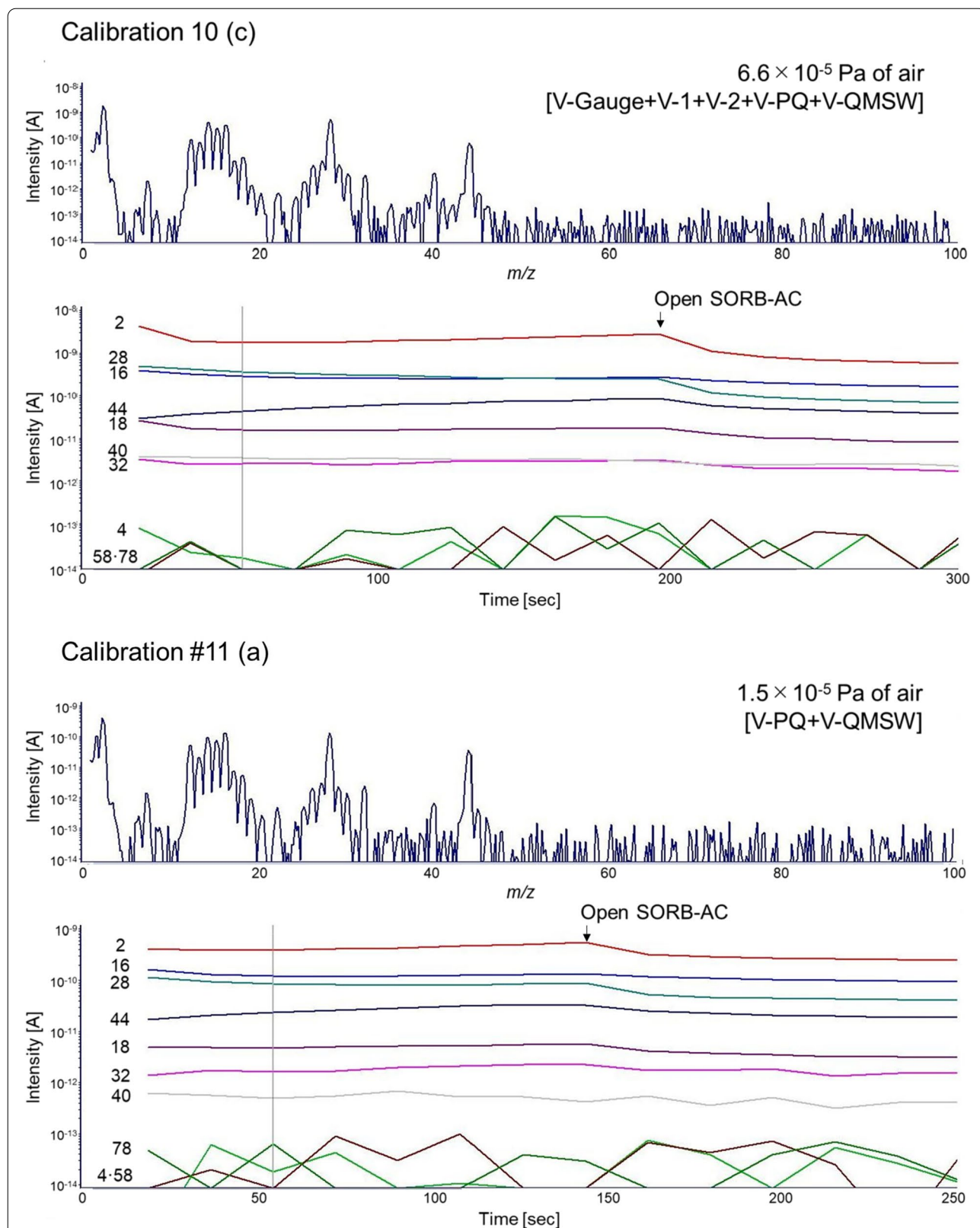


Fig. 8 Mass spectra for calibration gas measurements (calibration number of 10 (c), 11 (a) and 12) and a blank measurement. The mass spectra at ~55 s after gas introduction (at top) and intensities of representative m/z spectra with time (at bottom) are shown. The amounts of calibration gases (the terrestrial atmosphere) and the areas to which the calibration gases were introduced during mass spectrometry are indicated. Other calibration conditions for the measurements are summarized in Table 3

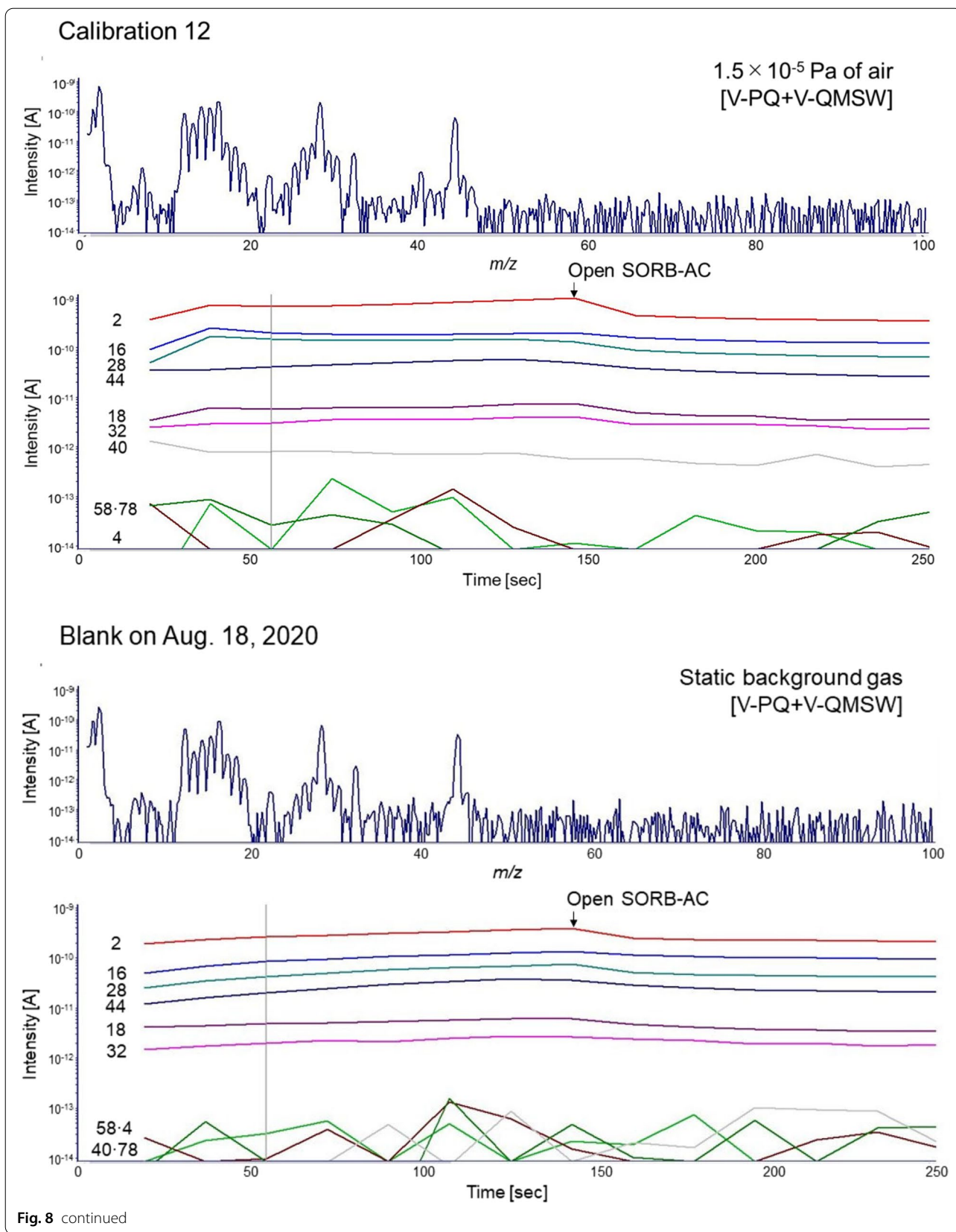


Fig. 8 continued

including isotope ratios must be determined using gases collected in the bottles in more detail.

The analytical procedure at the QLF in Woomera

An outline of the analytical protocols for extraction, storage, and measurements of volatile gases from the Hayabusa2 sample container is shown in Fig. 9. After the recovery and safety check operation of the SRC, the sample container is taken out of the SRC at the QLF in the WPA. The outside of the container is wiped with acetone and ethanol. The container is mounted on the ICF356 flange via a metal O-ring (Usui Co. Ltd., U-tightseal CS10033A100G50, whose outer diameter, inner diameter, wire diameter and groove depth are 57.6, 51.0, 3.3 and 0.5 mm, respectively). Then, it is connected to the GAEA through the vacuum flexible bellows labeled [V-FL] (Figs. 1 and 4). The air captured between the sample container and the GAEA is first evacuated. Overnight pumping and bake-out of the vacuum line of the GAEA are conducted, during which the sample container is not evacuated and heated. When pressure in the line becomes low enough for analyses

($\leq 10^{-7}$ Pa) and the connection flanges are checked by helium leak testing, gases are extracted through a hole made at the bottom of the container by moving a tungsten carbide needle attached on a linear motion feed-through (Fig. 1). The extracted gases are expanded in the volume of [V-Container + V-FL + V-Gauge] with the pressure measurement by the Pirani gauge (PG4 in Fig. 4). Then, the gases are expanded up to [V-PQ] and the gases in [V-PQ] are kept for measurements with the QMS (W). The fraction of [V-PQ] is to be 4.3% of the gases released from the container. The gases in the other volumes are expanded to the gas bottle area [V-NTTP + four gas bottles NT #1–#4]. The two valves for each bottle are closed to store the gases at room temperature. The four bottles should thus contain gases with the same amounts and compositions. The fraction stored in the four bottles is 80% of the extracted gases, meaning that each bottle contains 20% of the gases released from the container. The gases in [V-Container + V-FL] are held by closing the valve between [V-FL] and [V-Gauge], which are used for re-analysis later. The remaining gases in the line

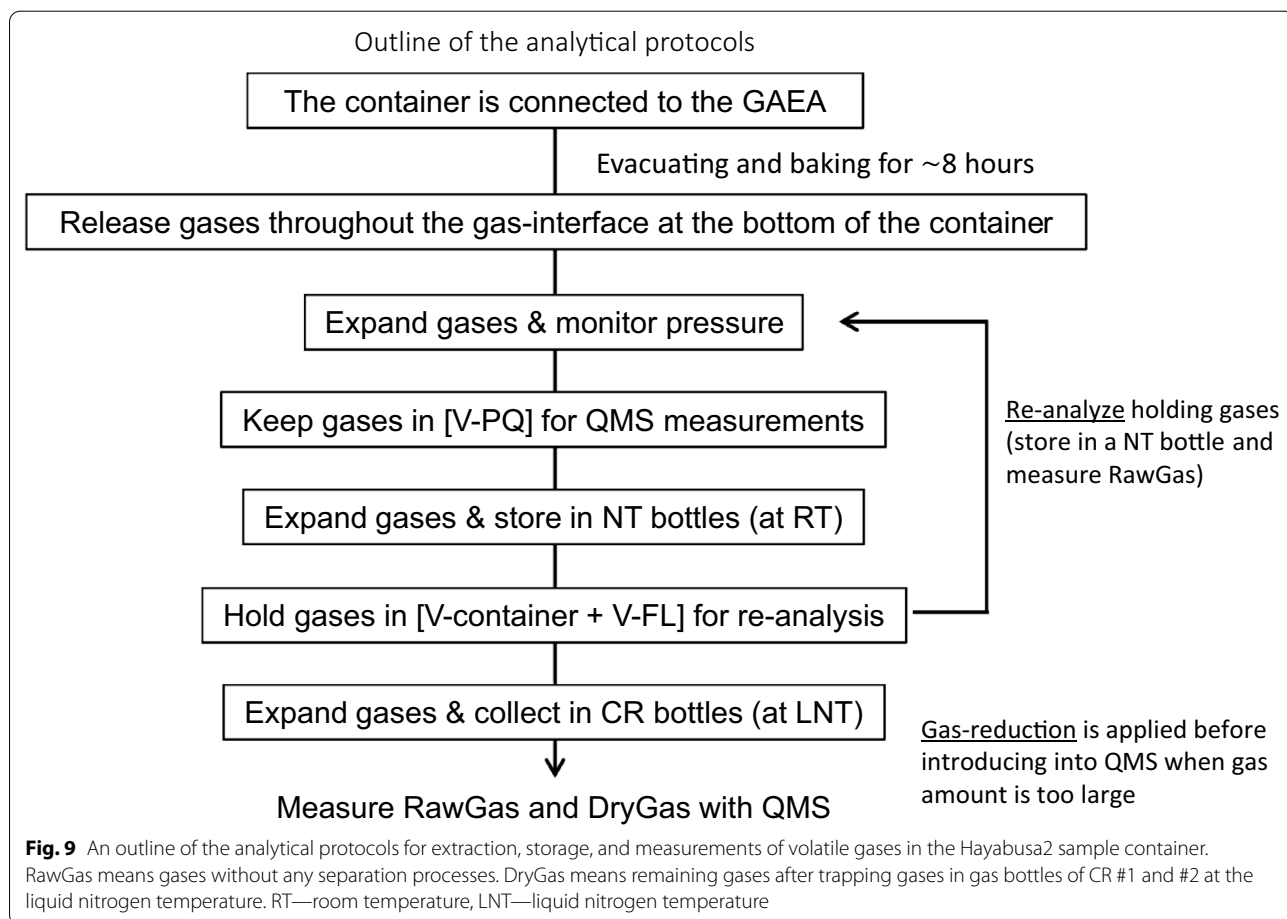


Fig. 9 An outline of the analytical protocols for extraction, storage, and measurements of volatile gases in the Hayabusa2 sample container. RawGas means gases without any separation processes. DryGas means remaining gases after trapping gases in gas bottles of CR #1 and #2 at the liquid nitrogen temperature. RT—room temperature, LNT—liquid nitrogen temperature

are expanded and sequentially trapped into two gas bottles, CR #1 and #2, cooled with liquid nitrogen at a 50%-height level from the bottom of the bottles for 2 min and 80%-height level for another 2 min. During gas trapping at CR #1 and #2, the pressure changes are monitored with PG4. Then, the gases stored in [V-PQ] are expanded to [V-Gauge + V-1 + V-2 + V-PQ] (the volume [V-Gauge + V-1 + V2] is evacuated in advance), and reduction procedures coupling the line areas (e.g., [V-PQ]/ [V-Gauge + V-1 + V-2 + V-PQ]) are applied before the QMS measurements until the pressure of the gases become lower than the maximum operatable pressure for the QMS (W) ($\sim 10^{-2}$ Pa). The fractions of the gases collected in the bottles and in [V-PQ] are shown in Table 2.

For the measurements with the QMS (W), mass spectra are obtained with the peak scan mode (m/z from 1 to 100 for nominal operation, and from 1 to 200 as an option) and the peak jumping mode for characteristic gases such as hydrogen, helium, carbon, methane, and water depending on gases detected using the peak scan mode. The procedure files for these measurements (recipes 1–5) are set in the operation software of the QMS (W) (Table 4). When gases are introduced into the QMS (W), the valve at the SORB-AC getter is kept close. The gases are firstly analyzed using the recipes 1 and 2 without SORB-AC and then using the recipes 3 and 4 with SORB-AC by opening the valve at SORB-AC. Peak scans of m/z from 1 to 200, the recipe 5, are performed when various intense peaks are observed at $m/z \leq 100$ and molecules with $m/z > 100$ are possibly

expected to exist. The obtained data are given as intensities in the unit of ampere (A), and then they convert to the gas amounts using the sensitivity of $520 \pm 100 \text{ cm}^3 \text{ STP/A}$ for ^{40}Ar and the sensitivity factor relative to ^{40}Ar of 1 ($\pm \sim 50\%$) as mentioned above. Because the intensities change with time during measurements in the QMS, the absolute amounts should be determined from stabilized data after gas input into the QMS, likely scan data after 50–120 s from the beginning of measurement with the recipe 1. On the other hand, although the absolute intensities often change with time depending on the gas pressure and the vacuum condition in a mass spectrometer, intensities relative to another likely remain nearly constant unless any condition-change such as opening the valve of SORB-AC. Therefore, relative intensities are calculated by averaging the data measured in peak jumping mode that have longer integration times; the results obtained by the recipe 2 (without SORB-AC) are used except for Ar isotope ratios. Data obtained by the recipe 4 (with SORB-AC) are more reliable for Ar because possible interferences of hydrocarbons (C_3 and C_3H_2) to ^{36}Ar and ^{38}Ar must be reduced.

The SRC-recovery was successfully performed by the Hayabusa2 capsule recovery team. After that, the extraction and measurements of volatile components in the sample container taken from the SRC were also completed at the QLF in Woomera as we planned. In this study, we have established a methodology using the GAEA, which is a promising prospect in combination with the successful recovery of Ryugu samples (Tachibana et al. 2022) and non-destructive analysis of solid

Table 4 Specifications and measured ranges of m/z for the QMS (Watmass) of the GAEA

Specification			
Filament	$\text{Y}_2\text{O}_3\text{-Ir}$		
Emission current	2 mA		
Electron energy	70 eV		
SEM voltage	900 V		
Integration time	32 ms		
Mode	SORB-AC ^a	Scan time ^b	m/z
Recipe 1. Peak scan	close	~ 120 s (~ 6 scan)	Peak scan from 1 to 100
Recipe 2. Peak jump	close	~ 90 s	Peak jump at 2, 3, 4, 14, 15, 16, 17, 18, 28, 29, 32, 34, 40, 44, 45, 46, 64, 66, 69, 78
Recipe 3. Peak scan	open	~ 90 s (~ 10 scan)	Peak scan from 1 to 44
Recipe 4. Peak jump	open	~ 90 s	Peak jump at 1, 2, 3, 4, 6, 12, 16, 18, 20, 21, 22, 24, 28, 29, 30, 32, 33, 34, 36, 38, 40, 44, 45, 46, 69, 78, 84, 132
Recipe 5. Peak scan		Optional	Peak scan from 1 to 200

A procedure plan used for the measurements of gases in the Hayabusa2 sample container

Measurements are performed sequentially from Recipe 1 to Recipe 4 in each gas fraction

Recipe 5 is also applied in the case where the gas amount is high

^a Gases are measured without SORB-AC and then with SORB-AC by opening the valve

^b Basic scan time depends on the gas amount and composition

samples (Yada et al. 2022; Pilorget et al. 2022). Results on the volatile compositions measured with the QMS and those in the gas bottles, collected at room temperature and with liquid nitrogen, will be reported elsewhere (e.g., Okazaki et al., *Science Advances*, in revision).

Summary

Hayabusa2 collected surface materials from the C-type asteroid Ryugu to understand the origin and evolution of the asteroid and the Solar System as well as to investigate primitive solar system materials. The collected samples were stored in the sample container, which was sealed by the metal-to-metal sealing system. In order to extract and measure volatile gases in the sample container without exposure to the atmosphere, we designed and constructed the system of ultra-high vacuum line equipping the interface with the sample container, the gas-storage bottles, and the QMS in the configuration of GAEA (the GAs Extraction and Analyses system). The system was set at ISAS/JAXA for pre-operation and training. After bake-out of the system and calibration measurements using terrestrial atmosphere, (i) nominal vacuum pressures were $\leq 1 \times 10^{-8}$ Pa and $\leq 1 \times 10^{-6}$ Pa at the evacuation lines and gas extraction area, respectively, and (ii) the sensitivity of ^{40}Ar was obtained to be $520 \pm 100 \text{ cm}^3 \text{ STP/A}$. Then, it was transported to Australia for the actual operation. The Hayabusa2 SRC with the Ryugu samples has landed in Woomera desert, Australia, on December 6, 2020. After recovery, the gas extraction, collection into the bottles, and measurements with the QMS were successfully operated using the GAEA. The analytical protocols are also presented here. The immediate gas extraction and handling technique described in this paper is likely applicable to future sample return missions.

Abbreviations

JAXA: Japan Aerospace Exploration Agency; ISAS: Institute of Space and Astronautical Science; WPA: Woomera Prohibited Area; QLF: Quick Look Facility; SRC: Sample Return Capsule; GAEA: GAs Extraction and Analyses system; HAWKS: HAYabusa2 gas collection With the Keyhole System; QMS: Quadrupole mass spectrometer; CCG: Cold cathode gauge; MG: Miniature ion gauge; PG: Pirani gauge; SEM: Secondary electron multiplier.

Supplementary Information

The online version contains supplementary material available at <https://doi.org/10.1186/s40623-022-01638-x>.

Additional file 1: Fig. S1. Design drawings of (a) gas bottle at normal temperature and (b) gas bottle for cold-trap. The former bottle and pipet have the volume of 750 and 40 cm^3 , and the latter bottle have 50 and 16 cm^3 , respectively. **Fig. S2.** Photographs of the GAEA at the ISAS/JAXA (Sagamihara) and the QLF (Woomera).

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Author contributions

YNM summarized data and prepared the manuscript together with RO, ST, and YT. RO and YNM made the design of the GAEA with contribution from YT, KS, ST, KY, SS, and HS. YNM, RO, and KS acquired preliminary experiments and calibration measurements. All authors contribute to the arrangement and improvement of the system, data reduction, and interpretation. All authors read and approved the final manuscript.

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Availability of data and materials

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

Declarations

Ethics approval and consent to participate

Not applicable.

Consent for publication

Not applicable.

Competing interests

The authors declare that they have no competing interests.

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