Trans. JSASS Aerospace Tech. Japan
Vol. 19, No. 4, pp. 477-484, 2021
DOI: 10.2322/tastj.19.477

A Feasible Study of *In-Situ* Measurements of Light Isotopes and Organic Molecules with High Resolution Mass Spectrometer MULTUM on the OKEANOS Mission

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(Received July 11th, 2019)

The OKEANOS mission utilizing the Solar Power Sail is one of the candidates of the strategic middle-class space exploration to the outer Solar System lead by the JAXA. The mission is planning to be launched in 2030's, and rendezvous for spectral observations and landing for in-situ measurements of light isotopes and organic molecules to a D or P type Jupitar Trojan asteroid in 2040's. The flagship instrument on board is a high-resolution mass spectrometry (HRMS) system together with suits of remote-sensing instruments. Through in-depth scientific observations, the OKEANOS mission will provide critical input to the key questions of (1) constraining planet formation/migration theories, and (2) inventory and distribution of volatiles in the Solar System. We have conducted experimental tests of a sample canister sealing by a metal seal knife-edge, sample canister-MULTUM test, gas chromatograph-MULTUM coupling test for organics, and H and N isotopic measurements in the atmospheric air. Current performances are (1) high mass resolution was 30,000 at m/z = 20, (2) sample canister system with a knife-edge metal seal kept 90 % of a released gas in 1 hour for Cu or Au gaskets with/without regolith, (3) error meets the required precision and accuracy for nitrogen isotopic measurement but for hydrogen. These experiment tests need to continue for our scientific proposes on the asteroids.

Key Words: OKEANOS Mission, Jupitar Trojan, *In-Situ* Analysis, Solar Power Sail, HRMS

Nomenclature

D	:	deuterium
EI	:	electron ionization
ESA	:	european space agency
ExoMars	:	exobiology on mars
GC	:	gas chromatograph
H	:	hydrogen
HRMS	:	high-resolution mass spectrometry
IOM	:	insoluble organic matter
JAXA	:	japan aerospace exploration agency
JFC	:	jupiter family comet
KBOs	:	kuiper belt objects
MOMA	:	mars organics molecule analyzer
MS	:	mass spectrometer
MSL	:	mars science laboratory
MULTUM	:	multi-turn time-of-flight mass spectrometer
m/z	:	mass-to-charge ratio
N	:	nitrogen
NASA	:	national aeronautics and space administration
O	:	oxygen
OCC	:	oort cloud comet
OKEANOS	:	oversize kite-craft for exploration and astronautics in the outer solar system
SPS	:	solar power sail

1. Introduction

The OKEANOS (Oversize Kite-craft for Exploration and AstroNautics in the Outer Solar system), which is also known as the Solar Power Sail, mission is one of the candidates of the upcoming strategic middle-class space exploration to the outer

Solar System lead by JAXA.[1] The mission is planning to be launched in 2030's, and rendezvous for spectral observations and landing for *in-situ* measurements of light isotopes and organic molecules to a D/P-type Jupitar Trojan asteroid about 2040's. Unique scientific instruments of a high-resolution mass spectrometry (HRMS) system together with suits of remote-sensing instruments will be on board.

Here we present scientific goals of *in-situ* analysis with the HRMS from the surface materials of a Jupiter Trojan asteroid. We summarized current developments of the HRMS system (MULTUM: multi-turn time-of-flight mass spectrometer[2]), on a lander including a sampling from the asteroid surface regolith, sample canisters with a heating (up to 600ºC) capability, a high-temperature pyrolysis oven for a decomposition of hydrous minerals and organics, and a gas chromatography system (optional) for analyzing organic molecules, as well as preliminary results of hydrogen and nitrogen isotopes and molecular analysis.

2. Scientific Goals

The OKEANOS mission is the first opportunity for a direct investigation of D/P type asteroid. Analyses of light isotopes and molecules of materials on a Trojan asteroid with the HRMS may permit deciphering if the Trojan bodies originate from the cometary reservoir or share similarities with asteroids (or meteorites) from the inner Solar System. Especially isotopic analysis may provide insight into the migration model of giant planets (Jupiter and Saturn) at the early Solar System. Key science questions of the OKEANOS mission are (1) constraining planet formation/migration theories, and (2) inventory and distribution of volatiles (water and organics) in the Solar System.

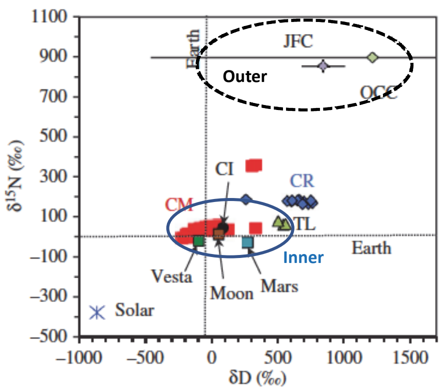

Fig. 1. Comparison of the estimated bulk H and N isotopic ratios of the major inner and outer Solar System bodies and carbonaceous chondrites that modified from Alexander (2017).[5]

2.1. Constraining planet formation/migration theories

The static model of the Solar System formation suggests that the Jupiter Trojan asteroids are mainly survivors of building blocks of the Jupiter system,[3] while planet migration models (e.g., Nice model) claim that the Jupiter Trojan asteroids are intruders from outer Solar System regions after the migration of the giant gas planets settled.[4] Thus, Jupiter Trojan asteroids potentially play an important role in understanding of the planetary formation and migration models. In case of the former scenario, the Trojans show similarities to primitive main belt asteroids (C or B type), and the later scenario, the Trojan asteroids show similarities to comets (Kuiper belt objects, KBOs). Hydrogen and nitrogen isotopic compositions in materials may help to decipher these scenarios (Fig. 1).

2.2. Inventory and distribution of volatiles (water and organics) in the Solar System

As the only sample for laboratory experiments from D/P type asteroid is the Tagish Lake meteorite (an ungrouped carbonaceous chondrite), our knowledge about the D/P type asteroids is very limited.[6] Consistent with aqueously-altered nature of Tagish Lake, main belt D type asteroids show 3 µm-band absorptions which indicates phyllosilicate OH, but Jupiter Trojans do not show the 3 µm band.[7] Thus, we might expect the very early stage of aqueous alteration and/or water ice (at subsurface) from the Trojan samples.

What was origin and evolutional processes of extraterrestrial organic matter? Comets contain larger amount of organics than primitive asteroids, i.e., carbonaceous chondrites. Alexander and coworker hypothesized that insoluble organic matter (IOM) in various chondrites and possibly cometary refractory organics evolved from a common precursor, which has high H/C ratios and high δD.[8] Organic matter in Trojan asteroids could be the missing-link of organic evolution from comets to asteroids.

Hydrogen isotopic composition (D/H ratio) of water (H_2O) in the Solar System objects is interesting with respect to the origin of Earth's water. Oort cloud comets have higher δD, and a Jupiter family comet (JFC) 103/P Hartley 2 shows similar δD to the Earth's water but recent result from Rosetta mission shows that water in 67P/Churyumov-Gerasimenko comet has high δD.[9] On the other hand, δD of water in chondrites are estimated to be lower than that of the Earth's water.[5]

The stable isotopic compositions of H and N for volatiles in the Jupiter Trojan asteroid may give us an important insight into circulation, distributions and evolution of gas and solid materials within the Solar System. The differences between primitive asteroids and comets are, in general, comets have not aqueously altered, and enriched in heavy isotopes (D and ^{15}N) than primitive asteroids due to isotopic fractionations in cold environments (e.g., molecular clouds and the outer

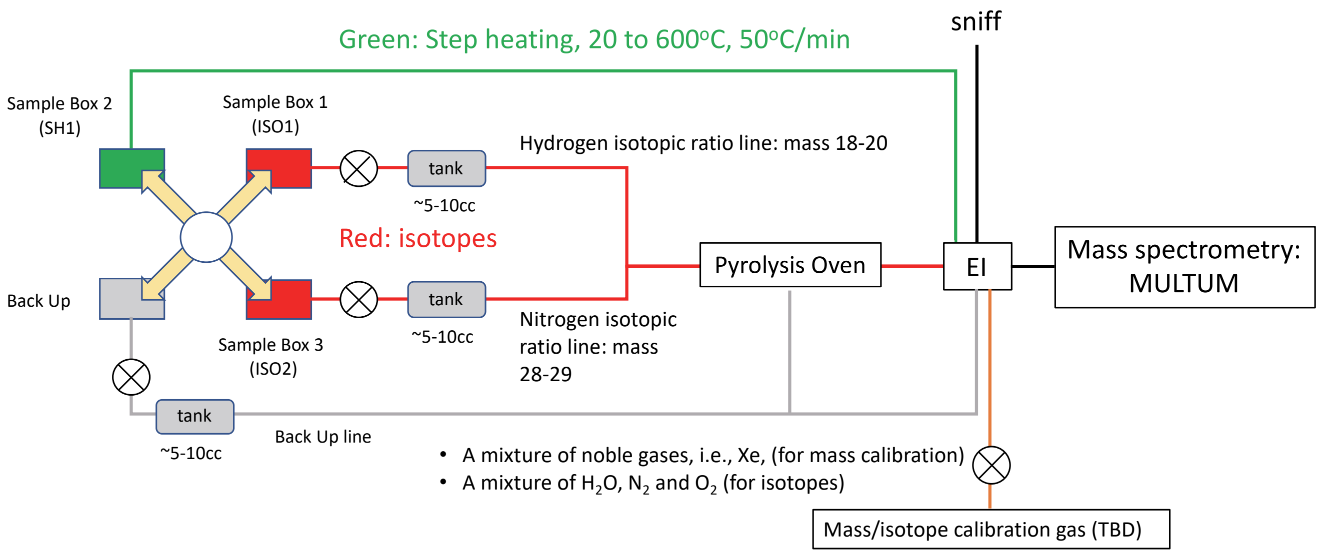

Fig. 2. Schematic diagram of the HRMS system in the OKEANOS mission.

protoplanetary disk) but not for primitive asteroids.[10-12] We, therefore, may find a genetic link between S, B, C-type asteroids (or meteorites) and comet (Fig. 1).

3. Developments of HRMS System

3.1. The HRMS

The HRMS is one of the flagship instruments on the OKEANOS, conducting critical measurements towards the scientific goals of the mission. D/P-type asteroids likely consist of dominant of organics (carbonaceous materials) and anhydrous silicates (hydrated silicates cannot be excluded), possibly with water (ice) in its interiors.[13] We plan to analyze volatile materials (at least 1 mg in each sample box) on the Jupitar Trojan, for their isotopic and elemental compositions using a MULTUM with a combination of three sample boxes having stepwise heating capability, a pyrolysis oven and EI (electron ionization) as an ion source. The schematic diagram of the HRMS system shows in Fig. 2.

This system allows us to measure H and N isotopic ratios and elemental compositions of molecules prepared by various pre-procedures including stepwise heating up to 600ºC, and high-temperature pyrolysis (~ 1,400ºC) in order to decompose the samples into simple gaseous molecules (e.g., H_2, CO, and N_2). The required mass resolution should be ~30,000 to analyze isotopic ratios (e.g., $H_2{}^{16}O$, $HD^{16}O$ and $H_2{}^{18}O$ for H and O isotopic measurements) in simple gaseous molecules. For determinations of elemental compositions of molecules/ions, mass accuracy of ~10 ppm is required to determine elemental compositions for molecules with *m/z* up to 300. In addition, 'sniff mode' which simply introduces environmental gaseous molecules into the MULTUM will be done by the system.

Operation hours of series of instruments on lander on the asteroid is demanded by mainly total budget of electricity (battery capacity). It is necessary to make a detailed plan for analysis utilizing the HRMS system and spectroscopic observations. The battery capacity on lander permits that the HRMS system is able to use 160 Wh in total. It is necessary to prepare a detailed plan to analyze surface materials on the Jupitar Trojan asteroid, for their isotopic and elemental compositions using the HRMS system. The more details of the analytical conditions and instrumentations are under developments.

3.2. Sample canister leak test

Sample canister for the OKEANOS mission will act as a container for a surface sample from the asteroid (~1 mg). It has a heating capability up to 600ºC with a stepwise capability (e.g., 50ºC/min) to release gasses in the sample. So, the sample canister should have a metal sealing by a Cu or Au gasket knife-edge system to not escape gasses, and a seal force up to a maximum force of 100 Newtons (N). It may be expected that dust particles (mainly regolith from the asteroid during sampling into the sample canister) could affect the seal performance worse. The regolith (a lunar regolith simulant) was used in this experiment, and they were uniformly distributed on the gasket.

The key is to maintain a released gas from the sample as much as possible in the canister during heating by an oven. We, then, have considered that requirement for sample canister to keep at least 80% volume of a released gas in the canister for 1 hour because it is necessary to supply a stable flow of the gas into the MULTUM for high-precision isotopic analysis. We, therefore, have conducted metal seal performance tests using an engendering model of sample canister (sample box) (Fig. 3) with Cu or Au gasket, different N_2 gas pressure (100 kPa and 20 kPa) and a commercial MULTUM (MSI Tokyo, infi-ToF).

We observed large leak of about 70% loss in 1 hour for Cu gasket and about 70% loss in 10 min for Au gasket if the regolith was on the gasket (Figs. 3(c) and 3(d)). On the other hand, if no regolith on the gasket, we found small leak of less than 10% in 1 hour for both gaskets (Figs. 3(c) and 3(d)). In

Fig. 3(e), we observed no leak for several hours without regolith on the Cu gasket and small leak of 20% loss in 3 hours under relatively low pressure (20 kPa) condition in sample canister.

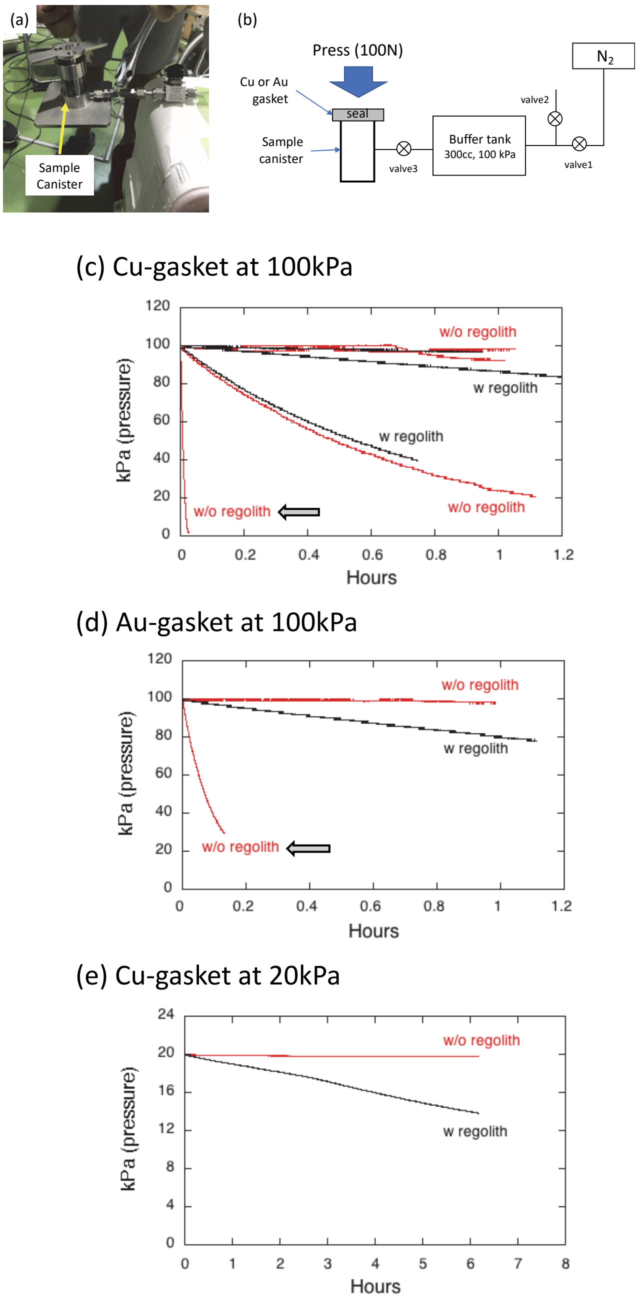

Fig. 3. A leak test for sample canister. (a) the engendering model of sample canister, (b) schematic diagram of the sample canister leak test (c) time-pressure profiles for Cu gasket seal test under 100 kPa, and (d) time-pressure profiles for Au gasket seal test under 100 kPa, and (e) time-pressure profiles for Cu gasket seal test under 20 kPa. Red solid lines are without regolith experiments (w/o regolith), and black solid lines are with regolith experiments (w regolith).

After each run, we have conducted surface observations of Cu and Au gaskets by a SEM (Hitachi SU1500 at Kochi Institute for Core Sample Research, JAMSTEC). Some experiments on Cu-gasket (arrow in Fig. 3(c)) and Au-gasket (arrow in Fig. 3(d)) show the inner pressure rapidly decrease without regolith on their surface of the gasket. The reason is most likely mechanical error of the seal system between gasket and knife-edge shaft; e.g., out of centering of the shaft on gasket. It is noted that some experiments without or with regolith on the gasket show similar inner pressure curves. This indicates that the current Cu or Au gasket knife-edge system is not optimized so the system needs to be improved in terms of mechanical assemble and materials of gasket and force.

3.3. Sample canister-MULTUM coupling test

As we described in the Section 3.2., the sample canister may have a small leak (Figs. 3(c)-(e)). We have conducted the test experiments of sample canister coupled with a commercial MUTLUM under vacuum condition (300-400 Pa) (Figs. 4(a)-(c)). We observed clear signals of ethanol which is way above from the background signals in six different experimental conditions with MULTUM (Fig. 4(d)). However, it was difficult to control a steady state of signal that is adequate for a high-precision isotopic analysis. Further experiments are necessary to make a control of gas flow rate into the MULTUM.

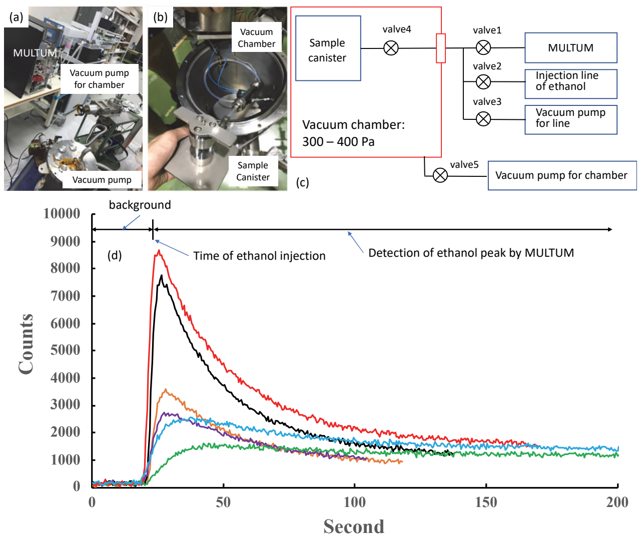

Fig. 4. Test experiment of the sample canister and a commercial MULTUM coupling under vacuum condition. (a) connection between sample canister and MULTUM under vacuum of 300 to 400 Pa, (b) sample canister in vacuum chamber, (c) schematic diagram of the sample canister and MULTUM coupling test, and (d) time-counts profiles of six different injections of ethanol measured by MULTUM.

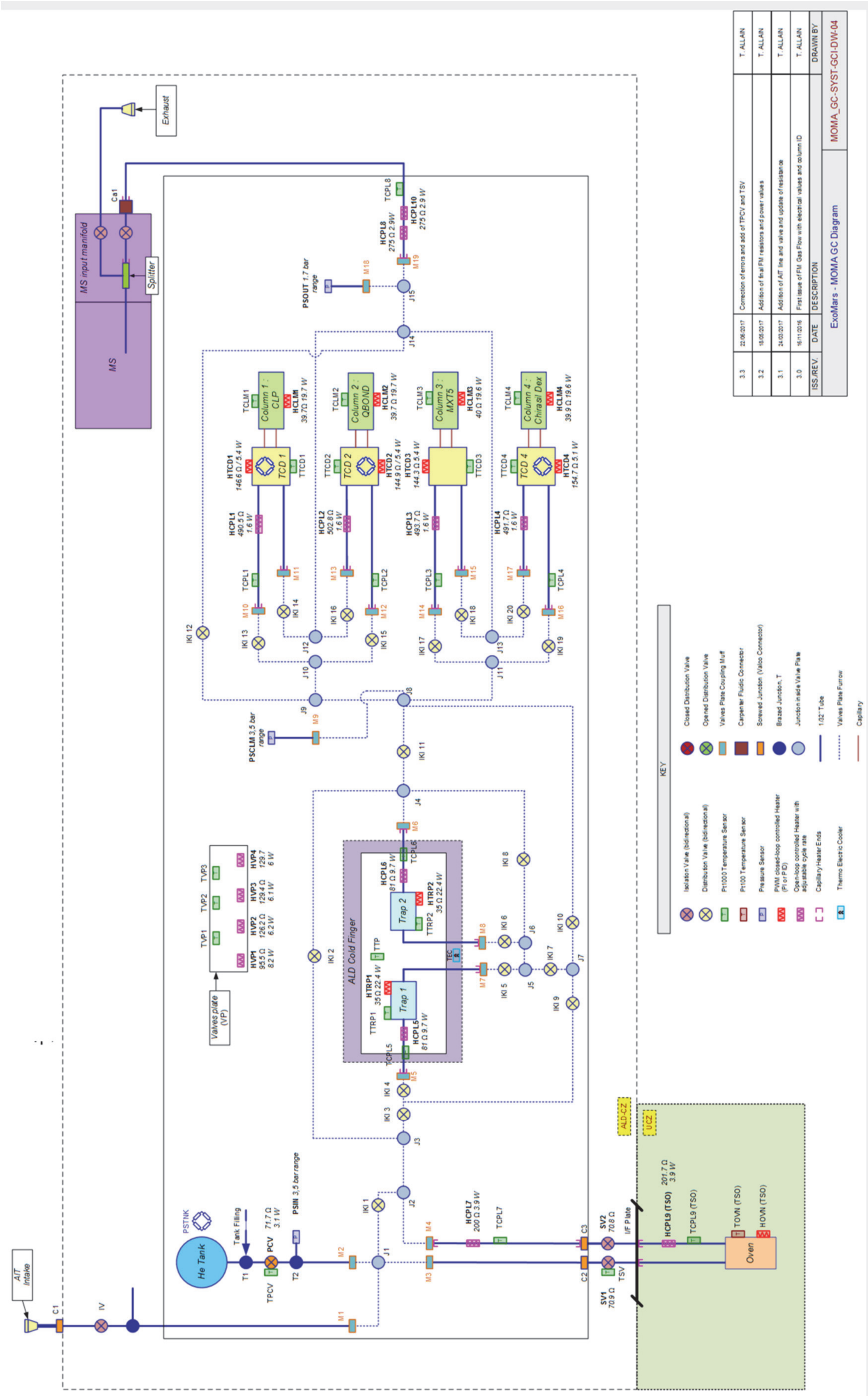

Fig. 6. A schematic diagram of the GC for MOMA on ESA/MSL mission as shown in Fig. 5 provided by Noel Grand.

3.4. GC-MULTUM coupling test for detecting organic components

In the first concept in the OKEANOS mission, a gas chromatograph (GC) coupled with the HRMS system have been proposed to characterize the organics at the surface of Jupiter Trojan asteroid. This system allows to analyze the composition of organic material obtained from the asteroid surface by the sampling system on the lander. Similar GC with mass spectrometer system on lander (e.g., NASA Mars Science Laboratory: MSL,[14]) was used to identify the molecules in Martian soils.

We have conducted a feasibility study of the GC-HRMS system at Osaka University utilizing an engineering model of GC developed for ESA/ExoMars missions (MOMA: Mars organics molecule analyzer),[15] with the MULTUM (Fig. 5). In Fig. 6, we show a schematic diagram of the GC for MOMA on ESA/MSL mission as shown in Fig. 5.

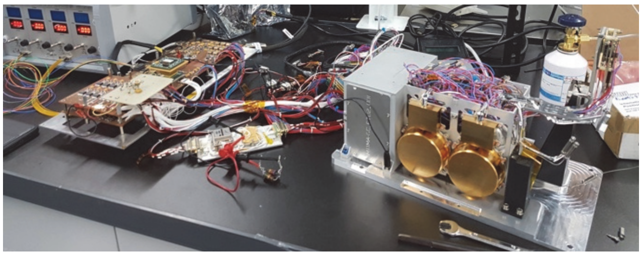

Fig. 5. The engineering model of GC for MOMA on ESA/MSL mission.

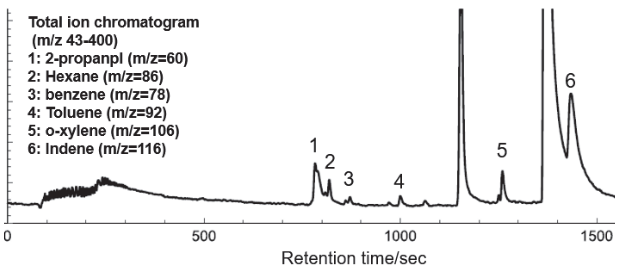

Fig. 7. Total ion chromatogram (TIC) of a mixture of six organic compounds measured by GC-MULTUM system.

A mixture of 2-propanol (*m/z* = 60), hexane (*m/z* = 86), benzene (*m/z* = 78), toluene (*m/z* = 92), o-xylene (*m/z* = 106) and indene (*m/z* = 116) was injected to a MXT5 column (20 m × 0.25 mm × 0.25 μm) connected to electron ionization (EI) source and a commercial MULTUM. The column temperature was set to 40°C for 19 min then ramped to 100°C with 20 °C/min, with helium flow at 1.20 mL/min. Each compound was successfully separated by the GC column and detected by MULTUM (Fig. 7).

3.5. Results of hydrogen and nitrogen isotopic measurements with HRMS

As shown in Fig. 1, the inner and outer Solar System materials show clearly differences in a combination of H and N isotopic composition.[5,10] So that analyses of these isotopes in materials on a Jupiter Trojan asteroid may permit deciphering if the Trojan bodies originate from the cometary reservoir as predicted by the giant planet migration models or share similarities with asteroids (or meteorites) from the inner Solar System.

We have conducted H and N isotopic measurements in an atmospheric air with a commercial MULTUM. The precision for H and N isotopic measurements are shown in Table 1, along with the required values. We defined the required precision and accuracy to address each scientific goal. Our results (standard error and reproducibility) meet the required precision and accuracy to archive our scientific purpose (e.g., Fig. 1) for nitrogen isotopic measurement but for hydrogen.

Table 1. Summary of H and N isotopic ratios in an atmospheric air and a water vapor.

Target Isotopes	environment	Measured Mass	Standard Error	Reproducibility	Required
Hydrogen	Water Vapor in Air	$H_2{}^{16}O$, $HD^{16}O$	δD: +-150‰	~ 150‰	+-100‰
Nitrogen	in Air	$^{14}N^{14}N$, $^{14}N^{15}N$	$\delta^{15}N$: +-20‰	~ 40‰	+-40‰

In Fig. 8, we show measured N isotopic ratios with time (~ 2 hours); red circles: 15 min accumulation, green circles: 3 min accumulation.

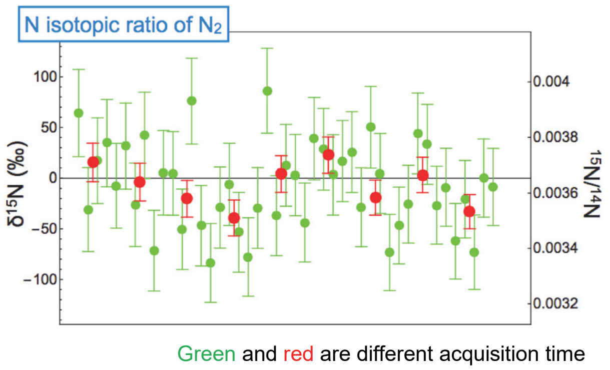

Fig. 8. Variations of N isotopic compositions with time (~2 hours).

4. Relationship to Other Missions

Collaboration with Lucy (multi-fly-by) of NASA Discovery mission will be enhanced an understanding of origin and nature of a Jupitar Trojan asteroid.[15] Detailed chemical analysis of single Trojan asteroid by the OKEANOS will support to understand the diversity among other Trojan asteroids by the Lucy mission. The sample return missions of C-type asteroid by the Hayabusa2 mission, B-type asteroid by the OSIRIS-Rex mission, and Martian moon, Phobos, by the Martian Moon eXploration (MMX) mission, will provide chemical and physical properties of different types of asteroids.[17-20] These may contribute better understandings of a relationship between comet and asteroid, planetary formation at the early Solar

System, and an inventory of volatiles. Phobos might be a captured D-type asteroid based on a spectral study by the OSIRIS (Optical, Spectroscopic, and Infrared Remote Imaging System on the ESA Rosetta spacecraft).[21)] Therefore, a returned sample analysis and remote sensing data by the MMX mission will provide a critical input to the *in-situ* HRMS analysis with the OKEANOS's *in-situ* analysis from Jupiter Trojan D/P-type asteroid.

5. Conclusion

The OKEANOS mission is a candidate for the upcoming strategic middle-class space exploration to the outer Solar System region. The mission is a combination of rendezvous and landing on a Jupiter Trojan asteroid (~20-30 km in diameter) utilizing a Solar Power Sail. We plan to analyze the asteroidal surface materials of a Jupiter Trojan asteroid using a multi-turn time-of-flight type high-resolution mass spectrometry (MULTUM) including an *in-situ* sampling system, a pyrolysis oven, as well as sniff mode capability.

We have conducted series of feasibility studies for developments of the HRMS system including a sample canister sealing by a metal seal knife-edge, sample canister-MULTUM coupling, GC-MULTUM coupling test for organics, and H and N isotopic analysis in atmospheric air. We have archived high mass resolution of 30,000 at m/z = 20 for H and N isotopic analysis utilizing a MULTUM. Best case scenario of current sample canister system with a knife-edge metal seal is able to keep 90 % of a released gas in 1 hour for Cu or Au gaskets with/without regolith. However, the system needs to be optimized in terms of mechanical assemble and materials of gasket and force.

For characterization of the organics, we have done a feasibility study utilizing GC (engineering models for the NASA/MSL and ESA/ExoMars) coupled to HRMS (MULTUM). We have obtained signals with six different organic compounds in their mixture. We have conducted hydrogen and nitrogen isotopic measurements in an atmospheric air with a MULTUM. Our results (standard error and reproducibility) meet the required precision and accuracy for nitrogen isotopic measurement but for hydrogen. We will continue to carry out these experiments for the detection of organics and for isotopes of H and N to archive our scientific proposes.

Overall, instrumental issues of sample canister, metal seal gasket, coupling between MULTUM and sample canister need to be improved at this moment, and for the detection of organics and isotopic measurements are close to meet to the mission requirement of the HRMS. It will be necessary to conduct combined experiments both instrumentations and analyzing of organics and isotopes utilizing the MULTUM system to fit on the mission requirements.

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